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Development of a Chemical Toxicity Assay for Pulp Mill Effluents

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A chemical analysis procedure was developed to measure within 1.5 hours compounds responsible for the toxicity of pulp mill effluents to fish. Analytical results for 113 samples of raw and biologically-treated bleached and unbleached kraft, sulphite and groundwood effluents were converted via toxic units into estimates of acute lethal toxicity. Agreement between 96-h median lethal concentrations calculated by this method, and values from bioassays of the effluents using rainbow trout was within 30% for 73% of the samples examined.

Biological treatment was highly effective in removing the known toxic compounds at pulp mills on the Willamette River, OR. and the Androscoggin River, N.H. and ME., based on estimates of toxicant loadings into and out of the biobasins.

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DEVELOPMENT OF A CHEMICAL TOXICITY ASSAY FOR PULP MILL EFFLUENTS

by

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FOREWORD

When energy and material resources are extracted, processed, converted, and used, the related pollutional impacts on our environment and even on our health often require that new and increasingly more efficient pollution control methods be used. The Industrial Environmental Research Laboratory - Cincinnati (IERL-Ci) assists in developing and demonstrating new and improved methodologies that will meet these needs both efficiently and economically.

This report describes the development of a chemical analysis procedure to rapidly analyze compounds responsible for the toxicity of pulp mill effluents to fish, and use of the results to estimate effluent toxicity as measured by standard laboratory bioassays. The report concludes that at present toxicity determined from chemical analysis can be a valuable supplement to, but not a substitute for, direct measurement by bioassay. The analytical technique was also used to measure the total loadings of detectable toxic constituents from four pulp mills on an east coast river and eight mills on a west coast river.

The report will be of interest to those engaged in identification and measurement of toxic materials in effluents. It is hoped that the approach used will promote further studies attempting to relate chemical characteristics of wastes to observed biological effects.

For further information on the subject contact the Food and Wood Products Branch, IERL, Cincinnati.

David G. Stephan
Director
Industrial Environmental Research Laboratory
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ABSTRACT

A chemical analysis procedure was developed to rapidly measure compounds responsible for the toxicity of pulp mill effluents to fish. The results were used to estimate effluent toxicity as measured by standard laboratory bioassays, and to determine loadings of various toxic compounds from all the pulp mills on two river systems.

The analytical procedure involved adsorption of toxic compounds from effluent onto Rohm and Haas Amberlite XAD-2 polymeric resin, elution of adsorbed constituents from the resin with ether and methanol, methylation of the extract, and quantitative analysis by glass capillary column gas chromatography. Analysis time was less than 1½ hours.

Results for 113 samples of raw and biologically-treated bleached and unbleached kraft, sulphite and groundwood effluents were converted *via* toxic units into estimates of acute lethal toxicity. Agreement between 96-h median lethal concentrations calculated by this method, and values from bioassays of the effluents using rainbow trout was within ± 30% for 73% of the samples examined. This degree of correlation is reasonable, bearing in mind the variations normally encountered in bioassay results. Calculated 96-h LC50's and those measured by bioassay differed by more than 30% for the other 27% of samples examined. At present, therefore, toxicity determined from chemical analysis can be a valuable supplement to, but not a substitute for, direct measurement by bioassay.

Samples were collected and analysed for five consecutive days from all pulp mills on the Willamette River, Oregon, and the Androscoggin River flowing through New Hampshire and Maine, to determine loadings of known toxic compounds into and out of the biobasins. Biotreatment at most of the mills was very effective in removing toxicants. Estimates based on theoretical dilution capacity of the receiving waters showed that secondary treatment was highly beneficial in protecting the rivers against the toxic effects of pulp mill effluents. Minimum dilution of pulp mill effluent discharged to the Willamette River during the study period (October, 1979) was 93-fold, assuming rapid and complete mixing. In the Androscoggin River, the minimum effluent dilution during the study period (February, 1980) was 17-fold.

This report was submitted in fulfillment of Grant No. R804977-01 by B.C. Research under the sponsorship of the U.S. Environmental Protection Agency. This report covers the period March 1, 1977 to February 28, 1980 and the work was completed as of August 31, 1980.

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SECTION 1

INTRODUCTION

In recent years, more than 30 organic compounds have been identified as contributors to the toxicity of pulp mill process effluents to fish (1-3). Chemical analysis procedures using gas chromatography (gc) have been developed for toxicants in the various waste streams (4-8), and toxicity of the effluents to fish has been estimated by summing toxic unit equivalents (9) of the measured toxicant concentrations (6-8). For many of the streams studied, good correlation has been obtained between the toxicity calculated from results of gc analyses and values obtained from acute lethal bioassays using rainbow trout.

As an extension of this work, the primary objectives of the study described in this report were to:

A. Develop a chemical analysis procedure for rapid measurement of the concentrations of toxic materials in bleached and unbleached kraft whole mill effluents, groundwood effluents and sulfite mill effluents.

B. Relate the chemical assay results to effluent toxicity measured in bioassays using rainbow trout.

C. Measure toxicant loadings from kraft whole mill effluents.

D. Measure toxicant loadings from groundwood and sulfite mills.

E. Make a preliminary assessment of the environmental significance of mill biotreatment systems for reducing toxic loadings in effluents.

F. Measure toxicant loadings from all mills on selected river systems.

The work was carried out in two phases; (I) development and evaluation of an analytical procedure for predicting effluent toxicity (objectives A, B); (II) use of the procedure for estimating toxicant loadings (objectives C-F).

SECTION 2

CONCLUSIONS

The analysis procedure devised in this project provides information in under 1.5 hours on the concentrations of individual major potential toxicants in pulp mill effluents. Acute lethal toxicity to rainbow trout measured by bioassay, and the toxicity values calculated from analytical results using the toxic unit concept, were in close agreement for a majority of primary- and secondary-treated effluents from softwood sulphite and groundwood mills, and softwood or hardwood bleached or unbleached kraft mills.

Analytical results provide information not available from bioassays on the nature of toxicants in mill effluents. This can be a guide to sources of toxic materials within the mill and an aid to identifying processes that need improved environmental controls.

However, since measured and calculated toxicity differed by more than 30% for 27% of the samples examined, the analysis method should be used to augment information from conventional bioassay and not as a replacement for such tests.

Well-operated biological treatment systems effectively reduced the concentrations of known toxic constituents in effluents to below acutely lethal levels.

Biotreatment of pulp mill effluents is most beneficial in protecting the Willamette and Androscoggin Rivers against acutely lethal effects of the discharges.

SECTION 3

RECOMMENDATIONS

Reasons for the lack of agreement in some cases between calculated and measured toxicity should be investigated. As a first step, the toxic components should be identified in effluents from mills that use hemlock, cedar, or hardwoods.

Since most research in this area to date has been carried out with cold water fish species, a similar study comparing calculated and measured toxicity using warm water test fish species would be of relevance and value to mills in the south.

Consideration should be given to using "standard" 96-h LC50 values of the individual toxicants to calculate the toxicity of effluents to a fish species. This would avoid problems of variations in bioassay results caused by the effects of seasonal and genetic differences on sensitivity between different stocks of the same species.

This study was concerned with acute lethal toxicity. Pulp mill effluents cause detectable sublethal effects in fish in the laboratory at concentrations of 0.1-0.2 of the 96-h LC50 (19). Studies should be undertaken to determine the degree of effluent treatment necessary to ensure that concentrations of toxicants in receiving waters are below these levels.

SECTION 4

MATERIALS AND METHODS

Phase I
Development and Evaluation of the Analytical Procedure

Pure samples of the toxic compounds were prepared using procedures described elsewhere (5-8).

Soluble material was removed from Amberlite XAD-2 adsorbent resin (2 liters; Rohm and Haas Co.) by washing an aqueous slurry in a glass column with methanol (500 ml), diethyl ether (4 liters), methanol (500 ml), deionized water (10 liters), 0.1% sodium hydroxide solution (1 liter), and deionized water (4 liters), in sequence. The resin was stored as a deionized water slurry in glass bottles.

Portions of the resin (12 ml) were added to glass columns (Figure 1) and rinsed with deionized water (3 x 15 ml).

CONDITIONS FOR ANALYSING STANDARD SOLUTIONS OF TOXICANTS

Preparation of Toxicant Solutions

Stock solutions of each toxic compound (25 mg/50 ml) were made in methanol. Mixed standards were prepared by pipetting appropriate volumes of the individual stock solutions into water (50 ml) to give the required concentrations. Ionic strengths of the solutions were varied by addition of saturated sodium chloride solution, and pH was adjusted with 1% sulphuric acid or 1% sodium hydroxide.

Adsorption of Toxic Compounds

An aqueous solution (50 ml) containing 0.1 - 0.5 mg of each of the toxic compounds was dispensed from a 250 ml separatory funnel through a column of XAD-2 resin at a flowrate of 15 ml/min. Air pressure was applied to the top of the column as required to maintain constant flow. The funnel was rinsed twice with deionized water and the washings were passed through the resin.

Elution of Adsorbed Constituents

Diethyl ether (15 ml) or 9:1 ether/methanol (15 ml) was added to the column and allowed to stand in contact with the resin for 10 min before eluting at 15 ml/min into a 125 ml separatory funnel. Two further aliquots of

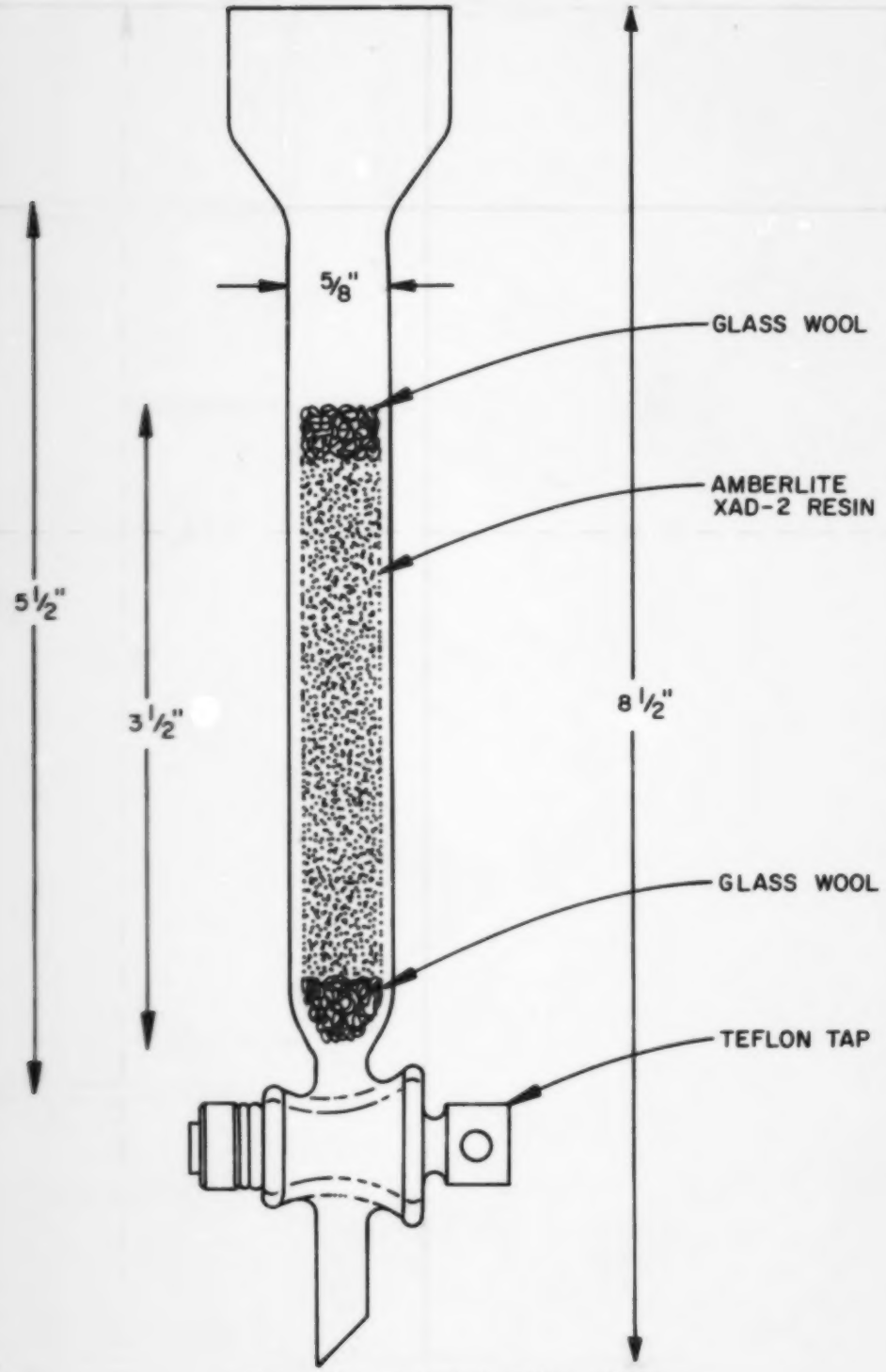

Figure 1. Resin adsorption column

solvent (10 ml) were passed through the column and collected.

Drying

Two procedures were examined for drying the solvent extract. In the first method, the water layer eluted with the solvent was drained from the separatory funnel, petroleum ether (BP 30-60°C; 15 ml) and anhydrous sodium sulphate (~2 g) were added to the organic phase, and the solution was shaken until clear. Solvent was drained from the separatory funnel through a Whatman No. 1 filter paper containing a small amount of anhydrous Na_2SO_4, and was collected in a 100 ml round-bottom flask. The funnel was rinsed with diethyl ether (2 x 5 ml) and the washings and filtrate were combined.

Another drying procedure entailed immersing the two-phase solution from elution of the resin in an acetone-dry ice bath for approximately 2 min. The organic layer was decanted from the frozen aqueous layer into a 100 ml round-bottom flask. The ice was thawed, washed with diethyl ether (2 x 5 ml), frozen again, and the organic extracts were combined.

Removal of Solvent

The extracts were concentrated by evaporating the solvent using: (a) a nitrogen purge at 30°C; (b) rotary evaporation under reduced pressure at 30°C; (c) distillation through a Vigreaux column, or (d) distillation using a Snyder (Kuderna-Danish) column.

Methylation

The concentrated extract was transferred to a 15 ml graduated centrifuge tube using ether. Methanol (0.5 ml) was added to extracts in which ether alone was the eluting solvent. An aliquot (1.0 - 2.0 ml) of a stock solution containing pentadecanoic (n-C_{15}), heptadecanoic (n-C_{17}), or nonadecanoic (n-C_{19}) acid in methanol (0.25 mg/ml) was added as an internal standard and the solution was methylated for 10 min with diazomethane generated from N-methyl-N-nitrosourea in an apparatus described by Levitt (16). The solution was then concentrated to approximately 0.1 ml by evaporation on a warm water bath.

NOTE: DIAZOMETHANE AND N-METHYL-N-NITROSOUREA ARE CARCINOGENS AND SHOULD BE USED WITH EXTREME CAUTION ONLY IN AN EFFICIENT FUME HOOD BY AN EXPERIENCED ANALYST WEARING DISPOSABLE GLOVES.

Gas Chromatographic Analysis

An aliquot (2-5 µl) of the methylated extract was injected into a Perkin-Elmer Model 3920 gas chromatograph containing an all-glass capillary system and flame ionization detectors. The column was 60 m x 0.25 mm, wall-coated with OV-101, temperature 240°C (isothermal), column pressure 42 psi helium, approximate split ratio 1:90, attenuation x10. Eluting peaks were recorded and measured using a Hewlett-Packard Model 3380A integrator, attenuation x1, area reject 10^3, slope sensitivity 0.03.

EFFLUENT SAMPLES

Samples (20 litres) of primary- and secondary- treated whole mill effluents were collected in rigid polyethylene containers from the mills listed in Table 1 and shipped by overnight road transport or by Air Express, usually arriving at the laboratory within 24 h of collection. Bioassays were carried out on the day of receipt, or on the day following receipt after overnight storage in tightly sealed containers at 2°C. Aliquots for analysis were adjusted with 10% NaOH to pH 10 and stored at 2°C for a maximum of 4 days. Analyses were normally undertaken within 48 h of sample receipt.

Procedure for Analysing Toxicants in Pulp Mill Effluents

The sample (20-50 ml of untreated effluent; 100 ml of biotreated effluent), adjusted to pH 9 with 10% NaOH solution and to a specific conductance greater than 2 mmho/cm using saturated NaCl solution, was passed from a 250-ml separatory funnel at 15 ml/min through a glass column (1.6 x 22 cm) containing Amberlite XAD-2 resin (12 ml), previously rinsed with deionized water (3 x 15 ml). The funnel was rinsed once with a small amount of saturated NaCl solution at pH 9, and residual effluent was expressed from the column using a stream of nitrogen. Adsorbed constituents were extracted by allowing 9:1 ether/methanol (15 ml) to stand in contact with the resin for 10 min before eluting into a 100-ml glass centrifuge tube at a flow rate of 15 ml/min. Additional ether/methanol (2 x 10 ml) was then passed through the resin to complete the extraction of adsorbed toxicants. Effluent trapped by the resin and eluted during passage of solvent was separated from the solvent layer by immersing the centrifuge tube in an acetone/dry-ice bath for 2 min. The organic layer was decanted from the frozen aqueous layer into a 100-ml round-bottom flask. The ice was thawed, washed with diethyl ether (2 x 5 ml), frozen again, and the organic extracts were combined.

Heptadecanoic acid (1 ml of a solution containing 0.05 mg/ml in methanol) was added to the solvent extract as an internal standard. The solvent was evaporated almost to dryness using a rotary evaporator, transferred using ether (6 ml) to a 15-ml graduated centrifuge tube, and concentrated to 2 ml by gentle warming on a water bath. Methanol (0.2 ml) was added and the solution was methylated with diazomethane for 10 min (16). The solution was evaporated to 0.1 ml on a warm-water bath, and aliquots (2-5 µl) were analysed by capillary column gc using a Hewlett-Packard Model 5830 FID instrument. Column specifications were: 16 m glass (0.25 mm i.d.) WCOT OV-101; initial temperature, 190°C, programmed at 1°C/min (20 min), 2°C/min (10 min) then 3°C/min to 250°C. Toxic compounds of interest were identified routinely by matching retention times with those of standards using a Hewlett-Packard Model 18850A integrator terminal with settings of attenuation 16, slope sensitivity 0.1, area reject 100, retention time window 2%. Identities of the constituents were authenticated in some samples by combination gas chromatography-mass spectrometry and in others by confirming that retention times coincided in samples "fortified" with added amounts of the pure compounds. Chlorinated guaiacol concentrations were checked by electron-capture gc

TABLE 1 MILLS FROM WHICH SAMPLES WERE OBTAINED FOR BIOASSAY AND ANALYSIS

Code	Mill Location	Type	Effluent Sample
A	British Columbia Coastal	Unbleached softwood kraft	Primary
B	Oregon	Bleached softwood kraft	Primary
C	British Columbia Interior	Bleached softwood kraft	Primary
D	British Columbia Interior	Bleached softwood kraft	Primary, secondary
E	British Columbia Interior	Bleached softwood kraft	Primary, secondary
F	British Columbia Interior	Bleached and unbleached softwood kraft	Primary, secondary
G	Mid-West U.S.A.	Bleached hardwood kraft	Secondary
H	East Coast	Unbleached low-yield sodium-base sulphite (softwood)	Primary
I	Ontario	Unbleached low-yield sodium-base sulphite (softwood)	Primary
J	Ontario	Bleached calcium/magnesium-base sulphite (softwood)	Primary
K	Maritimes Canada	Bleached ammonium-base sulphite (softwood)	Primary, secondary
L	N. Carolina	Bleached softwood and hardwood kraft	Primary, secondary
M	British Columbia Interior	Refiner groundwood	Primary, secondary
N	Washington	Bleached and unbleached softwood kraft; NSSC	Primary, secondary
O	Mid-West USA	Bleached hardwood kraft; hardwood NSSC	Primary, secondary
P	Washington	Bleached calcium-base sulphite (softwood)	Primary, secondary
Q	Oregon	Bleached ammonium-base sulphite (softwood)	Primary, secondary
R	Washington	Bleached ammonium-base sulphite (softwood)	Primary
S	Western Canada	Unbleached softwood kraft	Primary
T	Washington	Refiner groundwood (softwood)	Primary, secondary
U	Oregon	Unbleached softwood kraft; NSSC	
V	Oregon	Unbleached magnesium-base sulphite; refiner groundwood (softwood)	Primary, secondary
W	Oregon	Unbleached softwood kraft	Primary, secondary
X	Washington	Refiner groundwood	Primary, secondary
Y	Oregon	Bleached softwood and hardwood kraft	Primary, secondary
Z	Washington	Bleached softwood kraft and magnesium-base sulphite	Primary, secondary

(Hewlett-Packard Model 5710 instrument containing a glass column, 0.32 cm x 1.8 m, packed with 8% OV-1 on Chrom W (HP), 80-100 mesh; temperature, 175°C; carrier gas 95% argon, 5% methane; flow rate 20 ml/min) using aldrin as an internal standard. Samples from each mill were analysed before methylation to detect neutral compounds.

Recovery efficiency was measured using portions of the effluents to which known amounts of the toxic constituents had been added. Aliquots (4 or 8 ml) of stock solutions containing each toxicant in methanol (0.25 mg/ml) were mixed and diluted to 20 ml with methanol to give a solution containing 0.05 or 0.10 mg/ml of each compound. An aliquot (1 ml) of this solution was added to the effluent sample (20-100 ml), which was then processed through XAD-2 resin.

The gas chromatograph was calibrated daily using freshly made standard solutions of the individual toxicants.

Extraction of Toxic Compounds from Effluents Using Diethyl Ether

The effluent sample was adjusted to pH 4 with dilute HCl and extracted with ether (2 x 20 ml), and then to pH 2 and extracted again with ether (20 ml). The ether layers were combined, dried, concentrated, methylated and analysed in the same manner as ether/methanol extracts from XAD-2 resin.

BIOASSAY PROCEDURES

Static bioassays (3 or 6 liters) without solution replacement were used to measure the toxicity of effluents and pure compounds to juvenile rainbow trout (Salmo gairdneri). Washington and California State hatchery-reared fish acclimated to dechlorinated Vancouver City tapwater were used in the tests. Water quality characteristics included: pH 6.4, specific conductance 18 μmho/cm, alkalinity 4.5 mg $CaCO_3$/liter, EDTA hardness 4.8 mg $CaCO_3$/liter. Solutions were adjusted before dilution to pH 6.4 with 5% NaOH or 5% H_2SO_4 and maintained at that pH for bioassay by addition of McIlvaine's standard buffer solution [0.1 M citric acid (6.2 ml), 0.2 M disodium hydrogen phosphate (13.8 ml)]. Bioassay temperature was 11 ± 1°C, and dissolved oxygen was maintained above 9 mg/liter by gentle aeration of the test solutions through Pasteur pipettes. Fish loading was 0.5-1.5 g/liter.

The concentration to toxicant or effluent at which 50% of exposed fish survived for 96 h, i.e., the median lethal concentration (96-h LC50), or median tolerance limit (TLm_{96}), was calculated using a computer programme for the log-probit method of Litchfield (17).

Preparation of Standard Toxicant Solutions

Weighed amounts of dehydroabietic, abietic, pimaric, isopimaric, levopimaric, palustric, linoleic and linolenic acids were dissolved in methanol (5 ml); 5% NaOH solution (5 ml) was added dropwise and the solutions were made to 1 liter with dechlorinated water. To aid dissolution of oleic, monochlorodehydroabietic, dichlorodehydroabietic and epoxystearic acids, and of

trichloro- and tetrachloro- guaiacol, Tween 60 (30 mg) was added to the caustic methanol solution before diluting with water. Stock solutions were made to 10 liters with dechlorinated water, and adjusted to pH 6.4 with 5% sulphuric acid. Appropriate volumes were diluted and buffered at pH 6.4 for bioassay.

Phase II
Estimation of Toxicant Loadings from Operating Mills

Samples were obtained on five consecutive days from eight pulp mills on the Willamette River, Oregon, and on a separate occasion from four pulp mills on the Androscoggin River, Maine and New Hampshire (Table 2). Each day, polyethylene bottles were filled with biobasin influent (250 ml) and effluent (1 liter) taken from the regular 24-h composites collected by the mills. The samples were picked up between 9:00 a.m. and 1:00 p.m. and immediately stored in ice until processed later the same day.

Portions of the samples were passed in a field laboratory through GF/C filter paper under a vacuum created by a Cole Palmer Dynavac pump, to remove suspended solids. Aliquots of the filtered influent (50 ml or 100 ml) and effluent (100 ml or 200 ml) were adjusted to pH 9 with 5% NaOH solution and passed through a polyethylene column (Applied Science Laboratories) containing XAD-2 resin (10 ml) for analysis of dissolved toxicants. Columns containing the resin and adsorbed materials were capped with aluminum foil and refrigerated within 12 h of collection. In the Willamette study, the remainder of each sample was adjusted to pH 10 with sodium hydroxide pellets and frozen. In Maine, aliquots of the unfiltered influent and effluent were passed through XAD-2 resin on the day of collection, in the same way as for the filtered portions.

Aliquots of some samples were fortified in the field with known amounts of the toxic constituents, and then processed in the normal way to determine recovery efficiencies.

On the last two days of the study in Maine, additional samples of influent (8 liters) and effluent (20 liters) were collected in polyethylene containers for bioassay. Samples for bioassay from mills on the Willamette River were obtained subsequent to the 5-day study.

At the end of the week's sampling, the samples and resin columns were returned in ice to the laboratory. Materials adsorbed on the XAD-2 resin were recovered with 9:1 ether/methanol (35 ml), solvent was removed using a rotary evaporator, and the dried extracts were stored at -20°C until gc analyses were carried out. Extraction of the dissolved materials from all samples was completed within 6 days of arrival at the laboratory. Aliquots of the unfiltered samples, previously adjusted to pH 10 and stored frozen in the laboratory, were analysed for concentrations of total toxic constituents using the same procedures. Extracts of these samples were obtained for gc analysis within three weeks. Bioassays were commenced within 24 h of receipt in the laboratory, ie. not later than 4 days after collection at the mill.

TABLE 2 MILLS SAMPLED FOR TOXICANT LOADINGS STUDIES

Willamette River, Oregon

Unbleached kraft
Unbleached magnesium-base sulfite, refiner groundwood, and de-inking (2 mills)
Stone groundwood
Unbleached kraft/NSSC
Bleached kraft
Defibrated wood pulp
Bleached ammonium-base sulfite
----all softwood mills

Androscoggin River, Maine, New Hampshire

Bleached softwood and hardwood kraft
Bleached softwood and hardwood kraft, groundwood
Refiner groundwood
Bleached softwood and hardwood kraft, NSSC

Toxicant loadings were calculated from measured concentrations, and mean values of the figures provided by mill staff for daily effluent flows and pulp production.

SECTION 5

RESULTS

Phase I

EVALUATION OF ANALYSIS CONDITIONS FOR STANDARD SOLUTIONS OF TOXICANTS

Analytical conditions were varied to optimize accuracy and minimize analysis time for aqueous standard solutions of toxic constituents (11). Effects of the following variables on analytical accuracy and precision were studied:

1. pH and ionic strength of toxicant solution.
2. Use of ether or ether/methanol for elution.
3. Solvent evaporation technique.
4. Drying technique.
5. Internal standard for gc analysis.
6. Gc conditions.
7. Variation of gc retention time and response factor, with time and concentration.

Analytical procedures were evaluated for mixtures of the following compounds, which have been identified as important toxic constituents of various pulp mill process streams.

abietic acid
dehydroabietic acid
isopimaric acid
pimaric acid
sandaracopimaric acid
oleic acid
linoleic acid
linolenic acid
juvabione
juvabiol
dihydrojuvabione
pimarol
isopimarol
trichloroguaiacol
tetrachloroguaiacol
monochlorodehydroabietic acid
dichlorodehydroabietic acid
epoxystearic acid
dichlorostearic acid

Gas chromatographic conditions were varied until optimum separation of the compounds of interest was obtained, consistent with minimum total analysis time. Methyl esters of oleic and linolenic acids eluted with identical retention times and therefore could not be distinguished under the conditions used. Isopimarol was only partly resolved from methyl dehydroabietate and was omitted from subsequent studies of analytical accuracy and precision.

The first series of recovery efficiency tests involved comparison of different procedures for evaporating solvent from solutions of the compounds

in methanol. GC response factors of the various constituents relative to heptadecanoic acid added as internal standard were similar after concentrating the solution by rotary evaporation, nitrogen purge, or Vigreaux distillation (Table A-1) demonstrating that recovery efficiencies were similar by the three methods. Rotary evaporation was the fastest of the methods tested for removing solvent and was used in subsequent analyses.

Effects of pH and ionic strength (salting out) on adsorption by XAD-2 resin of mixtures of compounds from aqueous solution are shown in Table A-2. Diethyl ether was used to elute the adsorbed constituents from the resin, the drying agent was sodium sulphate, and heptadecanoic acid was used as the internal standard. Overall recovery efficiencies were better from solutions at pH 2 and pH 7 than at pH 10. Addition of salt did not improve the recovery of material (Tables A-2 and A-3). Solutions adjusted to pH 7 were used for subsequent tests.

Elution of adsorbed constituents using 9:1 diethyl ether/methanol gave better recoveries than elution using only ether (Table A-4). In conjunction with this solvent mixture, removal of water from the extract by freezing in an acetone/dry ice bath was a convenient and rapid drying technique.

Qualitative gc analysis showed that juvabione and heptadecanoic acid were not resolved under the conditions required for optimum separation of most other constituents. Pentadecanoic and nonadecanoic acids were examined therefore as possible internal standards. The gc peak for the methyl ester of pentadecanoic acid was well separated from other constituents, but recoveries of most compounds were apparently high (Table A-5), possibly as a result of impurity in the internal standard. In addition, methyl pentadecanoate eluted in a region of the gc trace which is frequently complex in samples of pulp mill effluents. However, methyl nonadecanoate eluted in a "window" in the gc trace between the fatty acid esters, juvabiones and chlorinated guaiacols, and the resin acids. Overall recoveries were good in tests using nonadecanoic acid as the internal standard, although high values were obtained for trichloroguaiacol and low recoveries were recorded for abietic acid (Table A-6). Results obtained earlier for diepoxystearic acid were unsatisfactory and this compound was omitted from standard mixtures in studies using nonadecanoic acid, since it is only a minor toxic factor in bleach plant caustic extraction streams and has not been detected in whole mill effluents. In a separate test, recovery of abietic acid from duplicate solutions passed through XAD-2 resin at pH 10 was 61% and 75%, compared with recoveries of 21% and 29% when the solution was at pH 7.

Variations in retention times and gc response factors over a 2-month period for the compounds analysed are shown in Table A-7. Changes occurred principally as a result of readjusting gc conditions after the instrument had been used for other analyses. Under continuous operation, retention times were reproducible to within 0.5%.

EVALUATION OF ANALYSIS CONDITIONS FOR EFFLUENT SAMPLES

Primary- and secondary- treated bleached and unbleached softwood kraft and sulphite whole mill effluents were obtained from the mills listed in Table 1. Initial samples were analysed using conditions found to be optimal for standard solutions of the pure toxicants, i.e. passage through XAD-2 resin at pH 7, conductance 5 mmho/cm. However, recoveries averaged only 60% from ali- Since in earlier studies (4-8), pH 9-10 was optimal for analysis of the toxicants in various pulp mill waste streams, the effect of pH on analytical accuracy was re-examined.

Preliminary tests showed that overall recovery efficiencies averaged 33% from effluent adjusted to pH 2, 60% at pH 7, and 84% at pH 10. Effect of the volume of sample processed was also investigated. Average recovery of toxicants from a 25 ml aliquot of clarified bleached kraft whole mill effluent was 86%, from 50 ml the average recovery was 88%, and from 100 ml the average recovery was 95%, based on known additions of the compounds. The reproducibility of the total analytical procedure, and of the gc analysis separately, was studied using multiple injections of two separate aliquots from the sample of effluent. For one aliquot, the total concentration of toxic compounds measured ranged from 6.26 to 7.25 mg/liter for three injections of the methylated extract; for the second aliquot the range was 5.74 to 6.54 mg/liter for four injections. Recovery of known amounts of constituents added to the extract averaged 103%, compared with an overall average recovery of 78% for the total analysis procedure for this sample. Typical gc traces for primary- and secondary-treated effluent samples are shown in Figures 2 and 3.

Direct solvent extraction of acidified effluents with diethyl ether, as proposed in another study (12) resulted in the formation of a stable intermediate emulsified layer, which probably accounted for the lower concentrations of most toxicants found compared with the resin adsorption method (Table A-8). Emulsions were also produced when two bleached kraft whole mill effluents processed through XAD-2 resin were acidified and extracted with ether to assess adsorption efficiency. However the results (Table A-9) indicated that less than 2% of the toxic material was not adsorbed by the resin. Because of uncertainty over the efficiency of ether extraction a further test was carried out using another sample, in which the percolated effluent was passed through a second column containing XAD-2 resin. Mean value of the concentration of material removed by the second column of resin was less than 3% of the concentration adsorbed by the initial column (Table A-10).

COMPARISON OF TOXICITY RESULTS FROM CHEMICAL ANALYSIS AND BIOASSAY

Preliminary results from analysis of toxicants in effluent samples indicated that the procedure showed promise of sufficient accuracy and reproducibility to warrant proceeding with the next stage of the study. This entailed comparing the toxicity of effluents to rainbow trout measured by bioassay, with values calculated from concentrations of the individual toxicants, using the toxic unit concept described below.

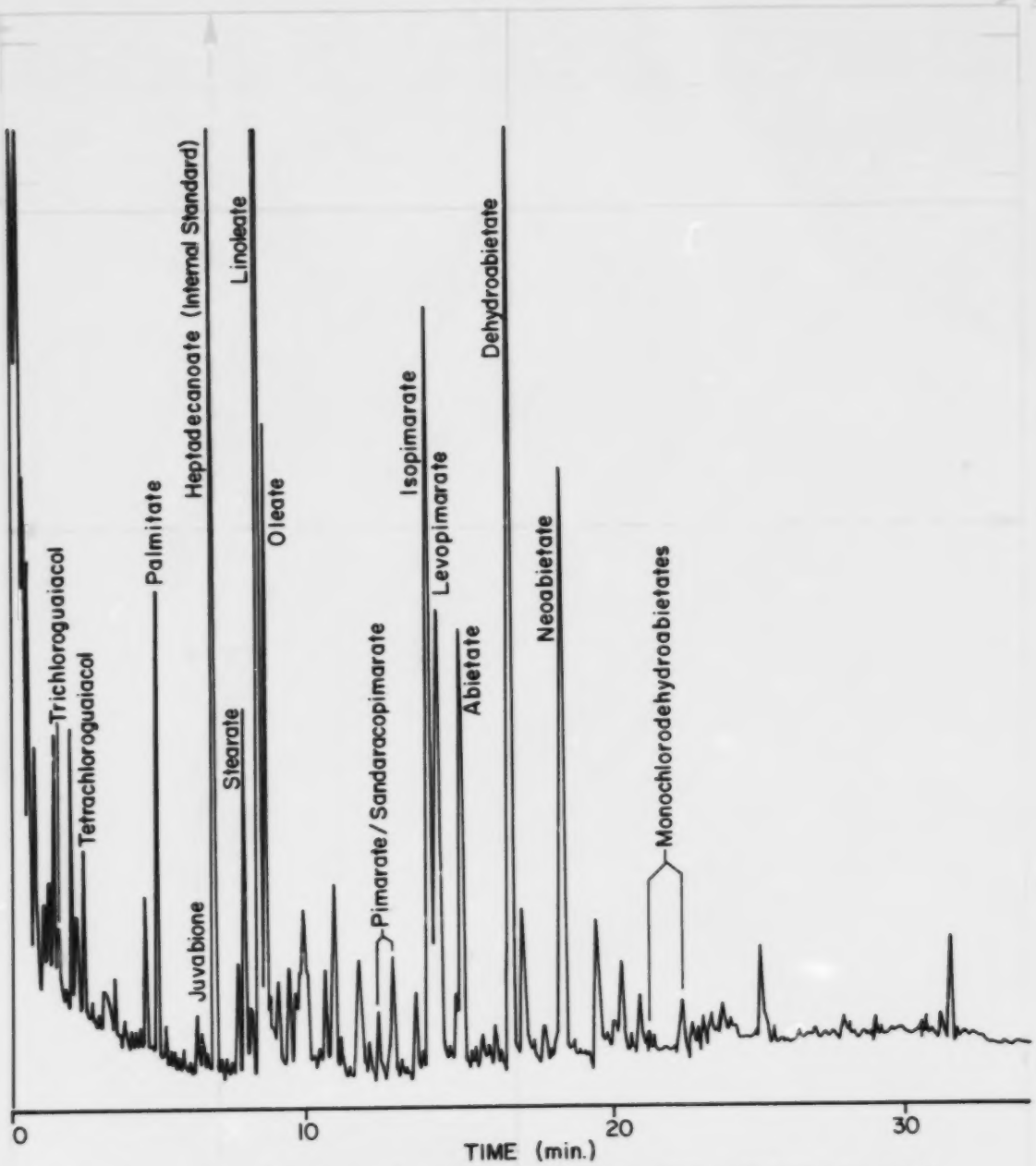

Figure 2. GC trace of methylated extract from primary-treated bleached softwood kraft effluent

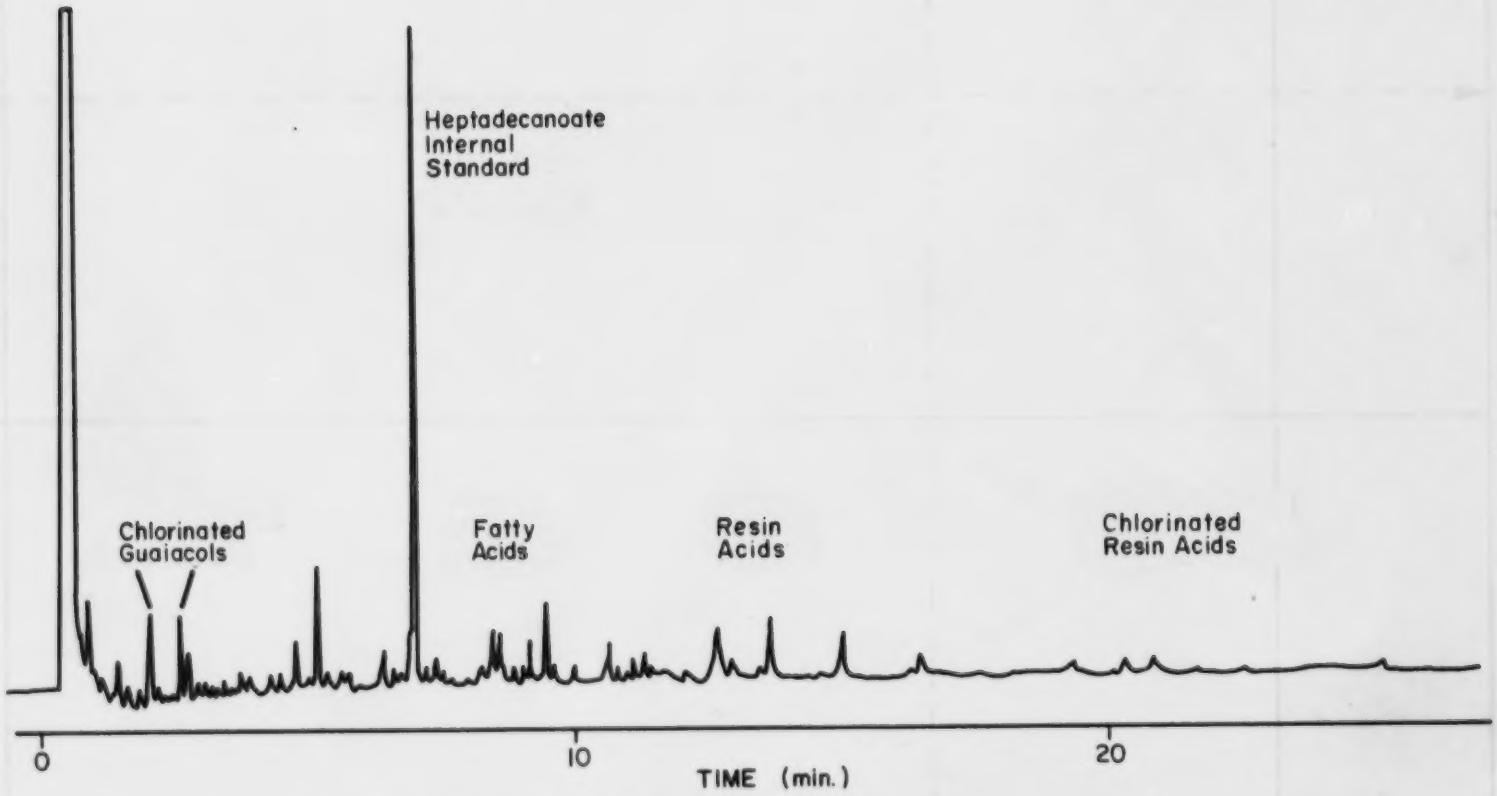

Figure 3. GC trace of methylated extract from secondary-treated bleached softwood kraft effluent

The strength of a toxicant in solution may be expressed as a multiple or fraction of the 96-h LC50 of the compound (9). The numerical value obtained is termed the toxic unit contribution. Thus:

$$\text{toxic units} = \frac{\text{conc. of a constituent in solution}}{\text{96-h LC50 of constituent}}$$

If there are no chemical or biological interactions that modify toxic effects, the toxic unit (t.u.) contributions of individual constituents in a toxicant mixture may be added to give a single value for the overall toxicity of the solution. This in turn can be translated into the 96-h LC50 of the solution by the relationship:

$$\text{96-h LC50 (\% by vol)} = \frac{100}{\Sigma \text{t.u.}}$$

A key requirement for the application of this concept is precise knowledge of the 96-h LC50 of individual toxicants. Toxic unit calculations for effluents analysed in the early phases of the study were based on 96-h LC50's measured during previous work (5-8). However, subsequent bioassays of the pure toxicants revealed differences in the response of rainbow trout stocks (Table 3, Figure 4). Therefore, toxic unit contributions were calculated for samples using 96-h LC50's obtained with fish from the same stock that was used to measure the effluent toxicity.

The analytical and toxic unit data in Tables B1-B12 give calculated and measured 96-h LC50's of effluent samples from Mills A-Z. Results (Table 4, Figure 5) are not corrected for analytical recovery efficiency. Agreement between 96-h LC50's estimated from analysis results (Tables B1-B12) and the values from bioassays was within 30% for 73% of 113 effluent samples examined. For approximately 10% of the samples, analysis results predicted a high toxicity value whereas bioassay indicated low toxicity, or vice versa.

Todomatuic acid and its methyl ester, juvabione, are not distinguished from each other in Tables B1-B12. From spot checks of unmethylated extracts by gc, most of this sesquiterpene was present in effluents as the acid. Under the gc analysis conditions used for effluents, juvabione in methylated extracts was separated from the heptadecanoate internal standard (cf p.14). No other neutral constituents were detected at significant concentrations in unmethylated extracts. Samples from some mills producing only unbleached pulp contained small amounts of compounds with the same retention times as trichloro- and tetrachloro- guaiacol on the WCOT OV-101 capillary column (flame ionisation detector), and on the packed OV-1 column (electron-capture detector). The concentrations are not recorded because of lingering doubt about the identity and origin of these compounds in unbleached processes.

Recovery efficiencies, measured by analyses before and after fortifying samples with known amounts of the toxic compounds, are shown in Tables C-1 and C-2. Mean values for the various compounds were in the range 80%-104%.

TABLE 3 BIOASSAY RESULTS FOR SOLUTIONS OF PURE TOXICANTS

Toxicant	96-h LC50 (mg/litre) with 95% confidence limits for juvenile rainbow trout						
	Stock #1; 6 weeks old	Stock #1; 12 weeks old	Stock #2; 6 weeks old	Stock #3; 6 weeks old	Stock #4; 16 weeks old	Stock #5; 4 weeks old	Previous Studies (5-8)
Dehydroabietic Acid	1.19 (1.10 - 1.28)	1.00 (0.94 - 1.07)	0.84 (0.72 - 0.97)	1.7	1.73 (1.61 - 1.83)	1.75 (1.67 - 1.85)	1.2
Abietic Acid	1.17 (1.04 - 1.31)	1.49 (1.31 - 1.71)	0.91 (0.84 - 0.97)	1.02 (0.96 - 1.09)	0.82 (0.78 - 0.88)	1.0 (0.95 - 1.05)	0.7
Pimaric Acid	0.67 (0.62 - 0.73)	1.14 (1.06 - 1.22)	1.23 (1.17 - 1.30)	0.70 (0.65 - 0.75)	0.61 (0.50 - 0.70)	0.49 (0.40 - 0.60)	0.8
Isopimaric Acid	1.05 (0.98 - 1.23)	1.00 (0.91 - 1.09)	0.52 (0.48 - 0.57)	0.54 (0.51 - 0.58)	0.35 (0.30 - 0.39)	0.32 (0.28 - 0.35)	0.4
Levopimaric Acid	0.97 (0.86 - 1.08)	1.05 (0.93 - 1.18)	0.63 (0.58 - 0.68)	1.02 (0.80 - 1.20)	0.77 (0.70 - 1.00)	0.66 (0.62 - 0.71)	-
Palustric Acid	0.64 (0.57 - 0.71)	0.60 (0.55 - 0.66)	0.48 (0.43 - 0.55)	-	-	-	0.5
Oleic Acid	3.60 (1.54 - 8.41)	>8.0	3.28 (2.77 - 3.89)	-	3.58 (3.24 - 4.14)	-	8.2
Linoleic Acid	2.57 (2.16 - 3.05)	4.47 (4.21 - 4.75)	2.59 (2.32 - 2.89)	-	2.47 (2.36 - 2.56)	-	2.0
Linolenic Acid	4.29 (3.80 - 4.85)	5.78 (5.45 - 6.13)	3.13 (2.88 - 3.41)	-	-	-	2.8
Trichloroguaiacol	0.79 (0.73 - 0.85)	1.02 (0.95 - 1.10)	0.71 (0.64 - 0.78)	0.79 (0.70 - 0.90)	0.81 (0.70 - 0.90)	0.74 (0.69 - 0.78)	0.75
Tetrachloroguaiacol	0.23 (0.21 - 0.26)	0.41 (0.36 - 0.45)	0.33 (0.28 - 0.39)	0.48 (0.43 - 0.55)	0.43 (0.40 - 0.48)	0.36 (0.33 - 0.39)	0.32
Monochlorodehydroabietic Acid	0.72 (0.68 - 0.77)	0.92 (0.83 - 1.01)	0.69 (0.62 - 0.77)	0.67 (0.63 - 0.72)	0.61 (0.55 - 0.67)	0.60 (0.56 - 0.64)	0.6
Dichlorodehydroabietic Acid	1.05 (0.93 - 1.20)	~3.2	1.16 (0.97 - 1.38)	0.52 (0.50 - 0.70)	0.34 (0.30 - 0.40)	0.24 (0.21 - 0.26)	0.6
Epoxystearic Acid	3.17 (2.81 - 3.58)	3.59 (3.19 - 4.04)	1.64 (1.51 - 1.78)	-	1.65 (1.54 - 1.74)	-	1.5
Neoabietic Acid	-	-	-	0.61 (0.50 - 0.70)	0.66 (0.61 - 0.72)	0.73 (0.65 - 0.92)	-

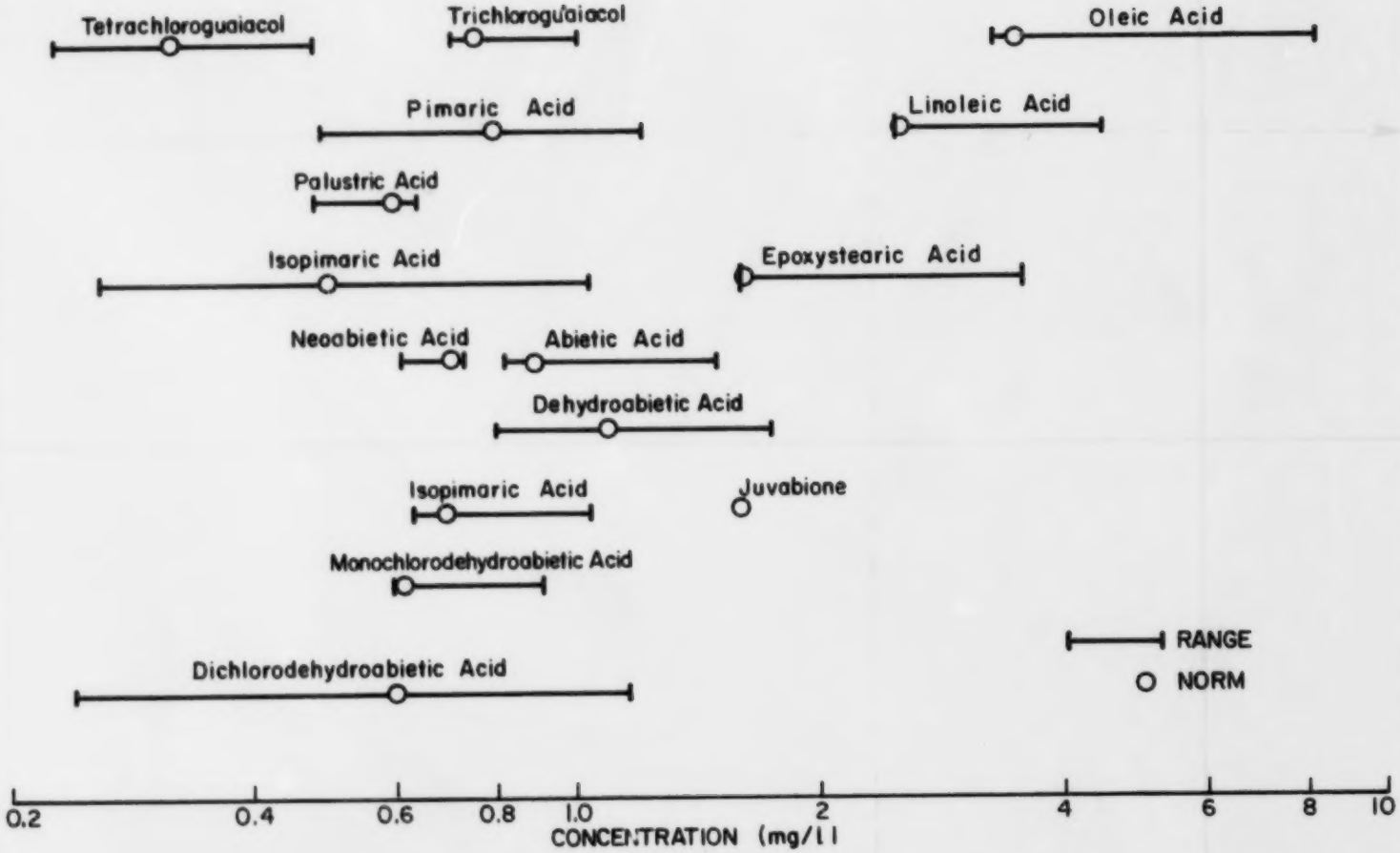

Figure 4. Ranges of toxicant 96-h LC50's

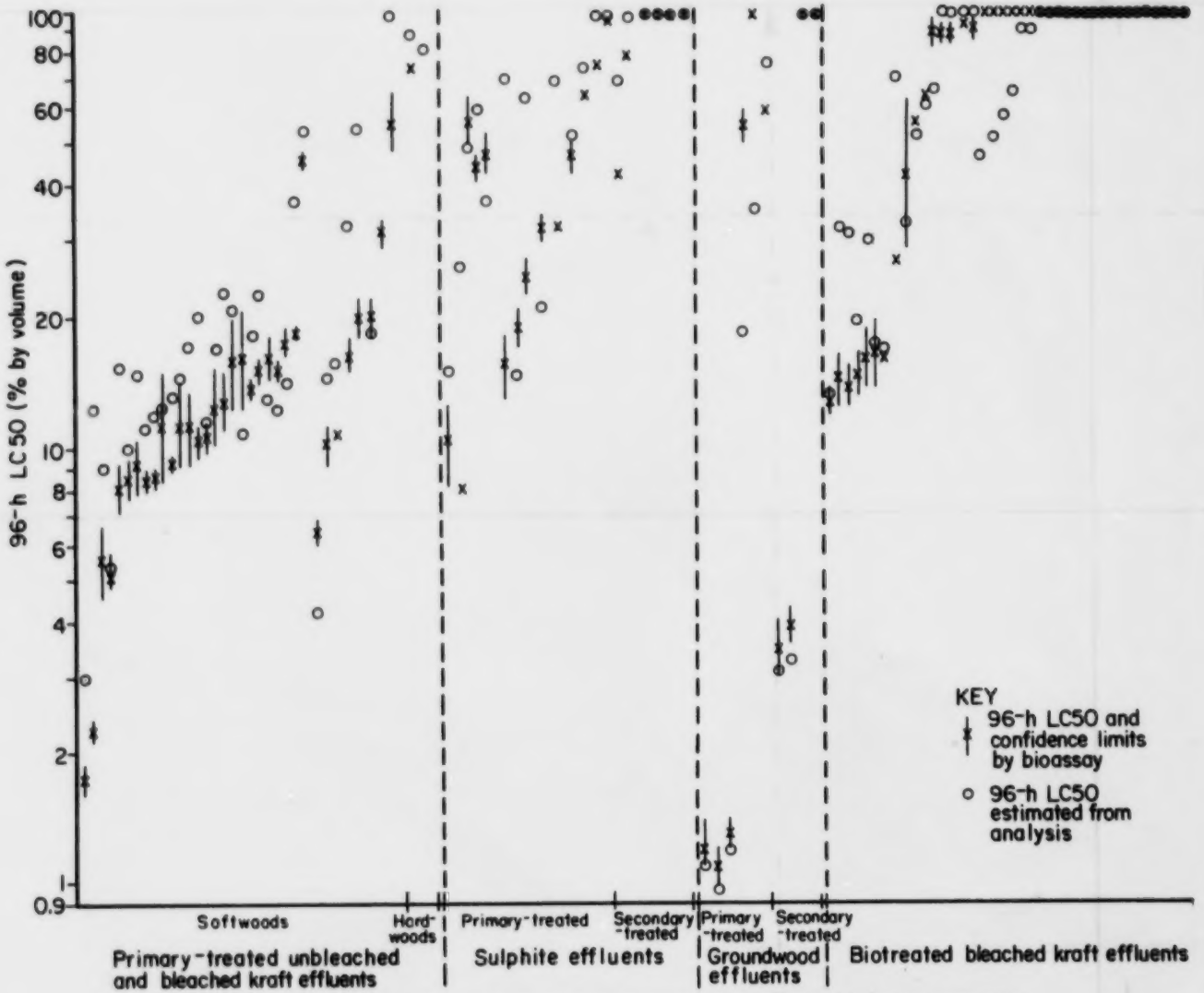

Figure 5. Comparison of calculated and measured effluent toxicities

TABLE 4 COMPARISON OF CALCULATED AND MEASURED TOXICITY VALUES

Effluent Type	Mill Code/Sample No*	96-h LC50 (% by vol)	
		Calculated from Analysis	Measured by Bioassay
Unbleached softwood kraft, primary-treated	A1	9.8	8.3
	A2	9.0	5.5
	F28	12.3	2.2
	F29	3.0	1.7
	S1	4.2	6.4
	U1	18.5	20.4
	W2	15.6	10.9
Unbleached softwood kraft, secondary-treated	U2	NT	NT
	W3	NT	NT
Bleached softwood kraft, primary-treated	B1	14.0	16.0
	D1	10.7	15.7
	D2	18.2	13.7
	D3	12.2	14.9
	D4	37.0	18.1
	D5	52.2	44.7
	D6	14.1	17.3
	E1	22.7	12.9
	E2	14.9	9.1
	E3	15.2	8.0
	E4	20.8	15.4
	E5	17.2	11.2
	F1	13.0	9.2
	F2	11.8	8.5
	F3	11.1	8.4
	F4	12.3	11.2
	F5	14.3	11.2
	F6	16.7	12.3
	F7	11.5	10.5
	F8	20.0	10.4
	F9	22.2	14.9
	F10	5.3	5.2
	N1	100.0	55.7
	N2	55.0	19.7
	N3	33.0	16.3
	N4	31.0	31.6

*Numbers are for samples collected from different effluent streams at a mill, or on different days from the same stream.

continued

TABLE 4(continued)

Effluent Type	Mill Code/Sample No*	96-h LC50 (% by vol)	
		Calculated from Analysis	Measured by Bioassay
Bleached softwood kraft, secondary-treated	D7	NT	NT
	D8	NT	NT
	D9	NT	NT
	D10	NT	NT
	D11	NT	NT
	E6	90.9	NT
	E7	NT	NT
	E8	NT	NT
	E9	NT	NT
	E10	52.6	NT
	E11	NT	NT
	E12	58.8	NT
	E13	13.2	13.1
	E14	17.2	16.3
	E15	17.5	16.6
	E16	31.2	14.2
	E17	30.3	16.4
	E18	20.0	15.1
	E19	32.2	14.6
	E20	33.3	42.5
	F11	NT	NT
	F12	66.7	90.0
	F13	NT	NT
	F14	71.4	27.4
	F15	47.6	NT
	F16	90.9	NT
	F17	100.0	NT
	F18	66.7	NT
	F19	71.4	57.4
	F20	52.6	57.4
	F21	100.0	NT
	F22	NT	NT
	F23	NT	92.7
	F24	NT	89.4
	F25	NT	89.4
	F26	100.0	94.9
	F27	62.5	64.8
	N5	NT	NT
	N6	NT	80.2
	N7	NT	>80.0
Bleached hardwood kraft, primary-treated	L1	90.0	84.0
	01	75.0	87.0
Bleached hardwood kraft, secondary-treated	G1	NT	NT
	L2	NT	NT
	02	NT	NT
Bleached mixed softwood and hardwood kraft; (1) primary-treated (2) secondary-treated	Y1	14.7	10.2
	Y2	NT	NT

*Numbers are for samples collected from different effluent streams at a mill, or on different days from the same stream.

continued

TABLE 4(continued)

Effluent Type	Mill Code/Sample No*	96-h LC50 (% by vol) Calculated from Analysis	96-h LC50 (% by vol) Measured by Bioassay
Bleached ammonium-base sulphite, primary-treated	K1	18.2	∿9.0
	Q1	NT	>60.0
	R1	NT	76.0
Bleached ammonium-base sulphite, secondary-treated	K2	71.4	∿45.0
	Q2	NT	NT
Unbleached sodium-base sulphite, primary-treated	H1	26.3	7.9
	I1	52.6	54.8
	I2	71.4	∿95.0
Bleached calcium/magnesium-base sulphite, primary-treated	J1	29.4	∿50.0
Bleached magnesium-base sulphite, primary-treated	Z1	71.0	15.7
Bleached magnesium-base sulphite and kraft; (2,3) primary-treated (4) secondary-treated	Z2	65.0	25.0
	Z3	76.0	65.0
	Z4	NT	NT
Unbleached magnesium-base sulphite and groundwood; (1) primary-treated (2) secondary-treated	V1	16.4	32.2
	V2	NT	NT
Bleached calcium-base sulphite and semi-chemical; (1,2) primary-treated (3,4) secondary-treated	P1	71.0	32.4
	P2	NT	97.0
	P3	NT	∿80.0
	P4	NT	NT
Groundwood, primary-treated	M1	1.1	1.2
	M2	0.96	1.1
	M3	1.2	1.3
	T1	77.0	60.0
	X1	18.5	55.4
	X2	36.0	100.0
Groundwood, secondary-treated	M4	3.1	3.3
	M5	3.5	3.9
	T2	NT	NT
	X3	NT	NT
Semi-chemical and NSSC, primary-treated	O3	15.0	18.0
	W1	53.0	47.6

NT - not toxic in 96 h

* Numbers are for samples collected from different effluent streams at a mill, or on different days from the same stream.

The target time for analysis, arbitrarily set at 1½ hours at the outset of the study, was attained (Table 5) during the latter part of the method evaluation stage, when effluent samples were being examined. Further reductions in analysis time do not appear to be practical with existing instruments.

Phase II
Estimation of Toxicant Loadings from all Mills on a River

WILLAMETTE RIVER

The analysis procedure developed and tested in Phase I of the study was used to measure the concentrations of potentially toxic compounds in the influents to and effluents from biotreatment ponds at all mills on the Willamette River, Oregon (Table 2). A random coding system is used in this report to preserve anonymity. The observation was made by industry representatives that some toxicants in mill effluents may be biologically inactive due to absorption onto suspended solids. As a consequence, concentrations of dissolved and total (i.e. soluble plus suspended) toxic constituents were determined. Results are shown in Tables D-1 to D-8. Table D-9 contains recoveries of toxicants added to certain samples in the field. For most compounds recovery efficiencies were in the range 80-100%.

Biobasins at all mills on the Willamette River were very effective in removing toxic compounds, both in the dissolved and suspended forms. The "total" concentration of each potential toxicant in secondary-treated effluents discharged from the mills rarely exceeded 10% of the 96-h LC50 of the compound and, except for Mill 4, was never higher than 20% of the 96-h LC50 for any toxicant. At Mill 4, the sum of the toxic unit contributions of all constituents analysed (suspended plus dissolved), approached or exceeded the 96-h lethal level in biotreated effluents. For effluents from the other mills on the Willamette River, the maximum total toxic unit value was equivalent to 0.4 of the 96-h LC50. Except in the case of oleic acid, suspended toxicants constituted more than 80% of the total figure in effluents, i.e. less than 20% of the toxicants were present in solution. Influents to the biobasins generally contained 20-40% of the fatty acids and 60-70% of the resin acids in the dissolved form (averages 36% and 55%, respectively). Higher concentrations of juvabione and its parent acid, todomatuic acid, were present in samples filtered on-site for determination of dissolved constituents than were found in unfiltered aliquots of the same samples analysed at a later date for total constituents. Similar anomalies occurred in a few samples for certain other constituents, notably oleic acid. The effect may have been caused by incomplete preservation of the unfiltered portion, since the discrepancy did not occur in the survey of mills on the Androscoggin River, where filtered and unfiltered samples were processed on the day of collection.

Separate samples of influent collected from each mill on the Willamette River after the main survey were analysed and tested for toxicity by bioassay before and after filtration. Nearly all the unfiltered samples were more toxic to fish than were the filtered portions (Table D-10) indicating that undissolved (suspended) toxicants in primary-treated samples were not entirely biologically inactive under laboratory bioassay conditions. Levels of sus-

TABLE 5 TIME REQUIREMENTS FOR CHEMICAL ANALYSIS OF TOXICANTS IN PULP MILL EFFLUENTS

Analysis Step	Time		
	Previous Method (5)	Target Time	Actual Time
Preparation of XAD-2 resin column	60 (75 ml resin)	5 (10 ml resin)	5 (12 ml resin)
Passing effluent through XAD-2 resin	60 (1000 ml sample)	10 (100 ml sample)	5 (50 ml sample)
Extraction of adsorbed toxicants	60 (1000 ml solvent)	10 (100 ml solvent)	15 (35 ml solvent)
Solvent evaporation and drying the extract	90	20	15
Methylation of extract	20	5	10
GC analysis	60	35	30
Calculation of results	60	5	5
TOTAL TIME	~7 hr	1 1/2 hr	1 hr 25 min

pended solids were too low to exert a "toxic" effect by blocking the gills of the fish. Correlations between toxicity measured by bioassay and the values calculated from analysis were good for influents from five of the eight mills, and mediocre to poor for the other three mills (coded 3,5,6).

Average loadings of the various types of potentially toxic compounds from each mill are shown in Tables 6 and 7. Averages for each compound are given in Tables D-11 and D-12. Total daily loadings discharged from all mills to the Willamette River were 2 kg chlorinated guaiacols, 52 kg C_{18} unsaturated fatty acids, and 71 kg resin acids. Approximately 20% of the fatty acids and 67% of the resin acids originated from Mill 4. Secondary treatment systems at the remaining mills removed 60-90% of unsaturated fatty acids, and 85-99+% of resin acids, based on "total" loadings in influents and effluents.

ANDROSCOGGIN RIVER

A companion study to that carried out on the Willamette River was done at the four pulp mills on the Androscoggin River (Table 2), which flows through New Hampshire and Maine. Concentrations of potentially toxic constituents in influents to and effluents from biotreatment ponds at the mills are shown in Tables E-1 to E-4. As in the Willamette River study, dissolved and suspended toxicants were removed with a high level of efficiency by biological treatment at all mills. The sum of toxic unit contributions for all the compounds analysed in biotreated effluent samples was generally 0.6 or less, i.e. 60% of the 96-h lethal level. For one sample from Mill I (7/2/80) and one from Mill IV (4/2/80) the combined "total" concentrations of toxicants in the treated effluent approached (0.9 of 96-h LC50) or exceeded (1.4 times 96-h LC50) the predicted lethal level. However, none of the secondary-treated effluents collected on the last two days of the survey was toxic to fish when tested in laboratory bioassays (Tables E-6). For most samples, the total (i.e. dissolved plus suspended) concentration of each potential toxicant in secondary-treated effluents was less than or equal to 0.2 of the 96-h LC50 value.

Between 65 and 90% of the resin acids in effluents from Mills I and IV, and approximately 40% of the resin acids from Mill II were in dissolved form. At Mill III, 90% of the resin acids were associated with suspended solids in the effluent. Dissolved unsaturated fatty acids comprised 15-85% of the total concentration depending on the mill. Primary-treated influents to the biobasins at Mills I, II and IV contained 60-100% of unsaturated fatty acids and resin acids in solution. Less than 10% of the total concentration of these compounds were in the dissolved form in influent at Mill III.

Toxicity calculated from analysis results for total (dissolved and suspended) toxicants generally correlated well with the values obtained by bioassays of influents and effluents (Tables E-5 and E-6). However, after filtration, measured and calculated LC50's differed considerably for dissolved constituents in influents at Mills II, III and on 7/2/80 at Mill IV. Reasons for these discrepancies are not evident at the present time. Most of the unfiltered influents were much more toxic to fish than were the filtered samples, demonstrating again that toxic components associated with suspended matter exert a biological effect in laboratory bioassays. The toxicant

TABLE 6 TOTAL LOADINGS (Kg/day)
WILLAMETTE RIVER MILLS

	Mill	Juvabione/ Todomatuic Acid	Fatty Acids	Resin Acids	Chlorinated Guaiacols
1	Influent	21.7	47.4	196	ND
	Effluent	0.08	5.0	5.4	ND
2	Influent	0.41	8.2	11.1	ND
	Effluent	0.58	3.3	1.7	ND
3	Influent	4.9	24.0	143	ND
	Effluent	<0.04	5.7	10.4	ND
4	Influent	7.9	31.0	273	ND
	Effluent	8.1.	10.2	47.7	ND
5	Influent	3.5	42.1	31.3	ND
	Effluent	<0.07	8.9	2.3	ND
6	Influent	2.5	6.8	13.9	11.4
	Effluent	0.05	1.1	0.05	1.9
7	Influent	12.0	77.7	249	ND
	Effluent	<0.04	17.8	3.8	ND
8	Influent	8.1	4.1	21.5	ND
	Effluent	0.10	0.34	0.08	ND
	Total to Willamette R.	8.9	52.4	71.4	1.9

ND - not detected

TABLE 7 TOTAL LOADINGS (g/tonne pulp)
WILLAMETTE RIVER MILLS

Mill		Juvabione/ Todomatuic Acid	Fatty Acids	Resin Acids	Chlorinated Guaiacols
1	Influent	41.2	89.9	371	ND
	Effluent	0.15	9.4	10.3	ND
2	Influent	2.9	58.5	79.1	ND
	Effluent	4.1	23.8	12.4	ND
3	Influent	4.1	20.4	121	ND
	Effluent	<0.04	5.7	8.2	ND
4	Influent	10.4	40.7	359	ND
	Effluent	0.39	13.4	57.2	ND
5	Influent	11.9	145	107	ND
	Effluent	<0.24	30.6	8.1	ND
6	Influent	5.8	15.9	32.4	26.6
	Effluent	0.12	4.0	0.12	4.7
7	Influent	19.5	126	405	ND
	Effluent	<0.06	28.9	6.2	ND
8	Influent	66.8	34.0	178	ND
	Effluent	0.79	2.8	0.68	ND

ND - not detected

composition of the grab samples collected for bioassay (Table E-5) generally differed from that of the regular 24-h composites (Tables E-1 to E4).

Average loading data for the individual compounds (Tables E-7 and E-8) are summarised for the different toxicant types in Tables 8 and 9. Total daily loadings discharged from all four mills to the Androscoggin River were 55 kg C_{18} unsaturated fatty acids, 115 kg resin acids, 21 kg tri- and tetrachlorinated guaiacols, and 2.7 kg chlorinated dehydroabietic acids. Mill IV discharge accounted for 56% of the resin acid loading to the river.

Secondary treatment at the mills removed 91-96% of the "total" unsaturated fatty acids, 92-97% of resin acids (except at Mill IV where resin acid removal was 80%), and amounts of the chlorinated guaiacols and chlorinated resin acids ranging from 50-90%.

TABLE 8 TOTAL LOADINGS (Kg/day)

ANDROSCOGGIN RIVER MILLS

	Mill	Juvabione/ Todomatuic Acid	Fatty Acids	Resin Acids	Chlorinated Guaiacols	Chlorinated Resin Acids
I	Influent	4.7	290	294	24.8	1.7
	Effluent	0.72	19.5	26.7	1.8	0.7
II	Influent	66.5	567	636	23.1	10.7
	Effluent	2.0	22.0	21.5	11.9	1.2
III	Influent	8.0	25.5	63.4	ND	ND
	Effluent	0.18	1.1	2.6	ND	ND
IV	Influent	28.8	133	299	20.3	8.6
	Effluent	11.0	12.5	64.4	7.3	0.8
	Total to Androscoggin River	13.9	55.1	115	21.0	2.7

ND - not detected

TABLE 9 TOTAL LOADINGS (g/tonne pulp)
ANDROSCOGGIN RIVER MILLS

	Mill	Juvabione/ Todomatuic Acid	Fatty Acids	Resin Acids	Chlorinated Guaiacols	Chlorinated Resin Acids
I	Influent	4.9	300	305	25.7	1.8
	Effluent	0.74	20.1	27.6	1.8	0.8
II	Influent	56.3	479	539	19.6	9.1
	Effluent	1.6	18.6	18.2	10.1	1.1
III	Influent	61.9	197	488	ND	ND
	Effluent	1.4	8.2	20.3	ND	ND
IV	Influent	48.4	224	503	34.2	14.4
	Effluent	18.5	21.0	108	12.2	1.3

ND - not detected

SECTION 6

DISCUSSION

Phase I

At the outset of the study, target time for the analysis procedure was 1.5 hours. To achieve this objective a micro-scale adaptation of existing techniques was needed. Because of the greater complexity of toxicant mixtures in whole mill effluents compared with the single process streams analysed previously, improved separation of the compounds of interest was required during gas chromatographic analysis.

Earlier work showed that XAD-2 resin can remove the toxic materials from large volumes of pulp mill effluent (4-8, 10). The semi-micro analysis procedure which evolved from these studies was scaled down to give a method that met the time requirements set initially in this project. Recovery efficiencies in the range 80-103% for toxicants added to effluent samples reflect a level of accuracy, compatible with other complex environmental analyses of organic constituents. In some samples stored for a few days low recoveries of levopimarate, accompanied by a high recovery figure for dehydroabietate, indicated that transformation of the labile levopimarate had occurred to give the chemically more stable dehydroabietate.

Resin acids were the predominant toxic constituents in primary-treated effluent samples from mills operating on softwood species. Levels of these compounds were generally, though not invariably, a good indicator of the effluent toxicity and the effectiveness of effluent treatments. Combined resin acid concentrations above 0.6 mg/liter usually were associated with effluents lethal to fish. Resin acids were important toxic contributors to primary-treated effluents from three mills using exclusively hardwoods, though the overall level of toxicity was lower than at softwood mills. Hardwood species are known to have a lower content of resinous extractives than do softwoods (18). None of the final biotreated effluent samples from American mills was toxic to fish in laboratory bioassays.

The extent of agreement between the calculated and measured toxicity of pulp mill effluents was similar to that obtained in a study relating the toxicity of sewage effluents to the concentrations of chemical constituents (14). There was a tendency for toxicity to be underestimated by analysis, although a few samples of biotreated effluent, which were not toxic by bioassay, contained levels of toxic compounds that were above predicted lethal thresholds. Reasons for the disparity between calculated and measured toxicity for some pulp mill effluent samples may include analytical or bioassay inaccuracies, particularly in earlier samples from Mills A-F when the analysis procedure was

being refined. Other discrepancies may have been due to the presence of undetected toxicants. For example, at sulphite mills the presence of sulphite ion, free sulphur dioxide, eugenols and, at ammonium-base mills, free ammonia (8) may have contributed to effluent toxicity. At one integrated mill, there was extensive use of dyes that may have influenced toxicity. The toxic constituents of effluents from mills using western hemlock have not been identified to date, and therefore there was a lack of agreement between calculated and measured toxicity of effluents from mills using high proportions of this species in the wood mix.

A further source of inconsistency in relating calculated toxicity to measured values may be biochemical interactions between toxicants that result in non-additive toxic effects. Also, constituents contributing less than 0.2 toxic units generally have been considered not to exert a measurable influence on acute toxicity (9). However, this may not be the case for toxicants in pulp mill effluents.

Clearly, causes underlying the disagreement between calculated and measured toxicity in certain samples must be understood in order to improve the congruency of analysis and bioassay results. Even so, the analysis procedure at present provides, in under 1½ hr, a useful estimate of toxicity to rainbow trout as measured by 4-day laboratory bioassay for a majority of pulp mill effluents.

Variations in bioassay results for the pure toxic compounds (Figure 4), and a published interlaboratory comparison of fish response to proposed standard toxicants (15) underscore a concern that bioassay data obtained for an effluent depend on the sensitivity of the fish stock used in the test. A chemical analysis approach to toxicity, coupled with "standard responses" of a fish species to each of the contributing toxicants, would avoid this uncertainty. The "standard 96-h LC50" for each toxicant could be the mean value computed from many determinations of the LC50 in waters at different temperatures, pH, dissolved oxygen, conductance, alkalinity etc., representing the differing characteristics of natural receiving waters. Alternatively, the "standard response" to each toxicant could be obtained for fish exposed in the receiving water to which the effluent is discharged.

This is not to suggest that chemical analysis of toxicants in pulp mill effluents should supplant bioassays, but rather that rapid monitoring of toxicity and its causes eventually may be done more readily on-site by gc analysis. For the present, however, bioassay is the only proven technique for measuring the toxicity of pulp mill effluents to fish.

Phase II

In Phase II of the study, loadings of the known potential toxicants were measured at all the mills on the Androscoggin and Willamette Rivers to assess in a preliminary way the environmental significance of mill biotreatment systems. Retention times of the various biobasins were from 6 hours to 14 days. All except one mill on the Willamette River and one on the Androscoggin River provided excellent removal of the toxic compounds analysed.

U.S. Geological Survey river flow data, and the effluent volumes provided by mill staff were used to estimate the maximum concentrations of various classes of toxic compounds that could conceivably occur in Willamette and Androscoggin River receiving waters. Results (Tables 10-13) are for the lowest dilution theoretically available at any point in the river (Appendix F), assuming the effluent mixes rapidly with river water, and no in-stream degradation of toxicants occurs.

Using the empirical relationship noted earlier that toxic pulp mill effluents usually contain above 0.6 mg/litre of resin acids, treatment systems on the Willamette River provided a 10-fold safety factor even for the "worst case" long-term minimum river flow (Table 10). Data in Table 11 demonstrate the benefit of secondary treatment in reducing the impact of pulp mill toxicants on the Willamette. The discharge of primary-treated effluents from all mills could result in acutely lethal levels of toxicants in the river under conditions of low river flow if there was no in-stream biodegradation.

Biotreatment at mills on the Androscoggin River reduced the concentrations of known toxic constituents to levels that give a 20-fold safety factor under long-term average river flow conditions (Table 12). Using the long-term minimum river flow figure the cumulative mill discharges could produce concentrations of toxic constituents that approach acutely lethal levels in the lower reaches of the Androscoggin River. This is a "worst case" circumstance, since it presumes that there is no self-purification in the river. Clearly, natural degradation does occur, and the possibility of such a situation arising is extremely remote. Furthermore, effluent samples from which the data are generated were collected under mid-winter operating conditions, when the average removal of resin acids was 91%. Treatment efficiency should improve in seasons when the air temperatures are higher. The environmental significance of good biological treatment at pulp mills on the Androscoggin River is evident from Table 13.

TABLE 10 ESTIMATED MAXIMUM CONCENTRATIONS (mg/liter) OF VARIOUS TOXICANTS IN WILLAMETTE RIVER FROM PULP MILL DISCHARGES

River Flow Datum	Juvabione, Todomatuic Acid	C_{18} Unsaturated Fatty Acids	Resin Acids	Chlorinated Guaiacols
October 15-19, 1979	0.001	0.02	0.02	0.0004
Long-term average	0.0005	0.006	0.007	0.0001
Long-term minimum	0.004	0.05	0.06	0.001

TABLE 11 ESTIMATED MAXIMUM CONCENTRATIONS (mg/liter) OF VARIOUS TOXICANTS IN WILLAMETTE RIVER IF PULP MILLS DISCHARGED PRIMARY-TREATED EFFLUENT

River Flow Datum	Juvabione, Todomatuic Acid	C_{18} Unsaturated Fatty Acids	Resin Acids	Chlorinated Guaiacols
October 15-19, 1979	0.09	0.1	0.44	0.002
Long-term average	0.03	0.04	0.17	0.0008
Long-term minimum	0.26	0.29	1.3	0.006

TABLE 12 ESTIMATED MAXIMUM CONCENTRATIONS (mg/liter) OF VARIOUS TOXICANTS IN ANDROSCOGGIN RIVER FROM PULP MILL DISCHARGES

River Flow Datum	Juvabione, Todomatuic Acid	C_{18} Unsaturated Fatty Acids	Resin Acids	Chlorinated Guaiacols
February 4-8, 1980	0.008	0.03	0.08	0.009
Long-term average	0.003	0.015	0.03	0.004
Long-term minimum	0.06	0.25	0.58	0.07

TABLE 13 ESTIMATED MAXIMUM CONCENTRATIONS (mg/liter) OF VARIOUS TOXICANTS IN ANDROSCOGGIN RIVER IF PULP MILLS DISCHARGED PRIMARY-TREATED EFFLUENT

River Flow Datum	Juvabione, Todomatuic Acid	C_{18} Unsaturated Fatty Acids	Resin Acids	Chlorinated Guaiacols
February 4-8, 1980	0.07	0.5	0.8	0.03
Long-term average	0.03	0.25	0.4	0.015
Long-term minimum	0.6	4.7	7.1	0.28

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TABLE A1 COMPARISON OF RECOVERY OF COMPOUNDS AFTER VARIOUS SOLVENT EVAPORATION PROCEDURES

Toxic Compound	GC Response Factor		
	Rotary Evaporation	Nitrogen Stream	Vigreaux Distillation
Trichloroguaiacol	2.00	2.00	2.01
Tetrachloroguaiacol	2.38	2.30	2.35
Linoleic Acid	0.80	0.82	0.83
Linolenic Acid	0.94	0.95	0.95
Pimaric Acid	1.00	1.02	1.03
Epoxystearic Acid	0.93	0.93	0.95
Isopimaric Acid	0.86	0.86	0.86
Dehydroabietic Acid	0.93	0.87	0.86
Abietic Acid	1.60	1.61	1.63
Diepoxystearic Acid	5.89	5.89	5.92
Dichlorostearic Acid	4.28	-	4.34
Monochlorodehydroabietic Acid	0.93	0.94	0.94
Dichlorodehydroabietic Acid	1.16	1.17	1.20

TABLE A2 RECOVERY OF COMPOUNDS AT pH 2, 7, 10 WITH AND WITHOUT SALT

Toxic Compound	% Recovery											
	pH 2				pH 7				pH 10			
	No Salt		+ Salt		No Salt		+ Salt		No Salt		+ Salt	
	2.1 mmho/cm		7.2 mmho/cm		4 μmho/cm		5.1 mmho/cm		22 μmho/cm		5.1 mmho/cm	
	1	2	1	2	1	2	1	2	1	2	1	2
Trichloroguaiacol	87	92	92	98	106	122	103	100	69	53	48	69
Tetrachloroguaiacol	103	110	93	94	(150)	105	94	95	-	44	52	47
Linoleic Acid	74	66	44	44	60	67	73	58	41	40	46	46
Linolenic Acid	74	66	71	61	60	65	59	50	46	46	46	46
Pimaric Acid	55	48	59	58	65	83	75	64	77	75	60	66
Epoxystearic Acid	102	104	80	87	70	80	82	83	32	30	28	27
Isopimaric Acid	52	46	54	54	65	81	74	59	81	78	64	70
Dehydroabietic Acid	71	72	86	90	74	98	82	79	72	71	67	71
Abietic Acid	49	36	31	31	37	-	56	(20)	82	67	52	58
Diepoxystearic Acid	(127)	(133)	(135)	(140)	68	65	97	94	(17)	(17)	(14)	(12)
Dichlorostearic Acid	84	74	60	53	69	55	67	63	32	35	36	52
Monochlorodehydroabietic Acid	53	47	59	49	72	83	80	71	75	72	63	62
Dichlorodehydroabietic Acid	48	46	54	38	77	75	68	61	74	72	60	58
Mean ± SD	71±21	66±25	65±19	63±23	68±15	82±19	78±14	73±17	62±20	56±17	53±12	57±13
Range	49-103	36-110	31-93	31-98	37-106	55-122	56-103	50-100	32-69	30-78	28-67	27-71

( ) outliers omitted from calculations of mean and range.

TABLE A3 EFFECT OF SALT ADDITION ON RECOVERY AT pH 7.0

Toxic Compound	% Recovery					
	No Salt		2 ml/200 ml		4 ml/200 ml	
	4 µmho/cm		5.1 mmho/cm		9.8 mmho/cm	
	1	2	1	2	1	2
Trichloroguaiacol	106	122	103	100	103	97
Tetrachloroguaiacol	(150)	105	94	95	98	95
Linoleic Acid	60	67	73	58	53	57
Linolenic Acid	60	65	59	50	39	40
Pimaric Acid	65	83	75	64	57	59
Epoxystearic Acid	70	80	82	83	77	84
Isopimaric Acid	65	81	74	59	56	56
Dehydroabietic Acid	74	98	82	79	74	71
Abietic Acid	37	-	56	(20)	50	52
Diepoxystearic Acid	68	65	97	94	92	96
Dichlorostearic Acid	69	55	67	63	52	52
Monochlorodehydroabietic Acid	72	83	80	71	(125)	119
Dichlorodehydroabietic Acid	77	75	68	61	93	107
Mean ± SD	68±15.6	82±19	78±14	73±16.7	75±25.9	76±25.1
Range	37-106	55-122	46-103	50-100	39-103	40-119

( ) outliers omitted from calculations of mean ± SD and range.

TABLE A4 RECOVERY EFFICIENCY USING ETHER/METHANOL FOR ELUTION COMPARED WITH ETHER ALONE

Toxic Compound	% Recovery							
	Ether		Ether/Methanol					
	1	2	1	2	3	4	5	6
Trichloroguaiacol	75	99	(148)	123	99	98	(132)	115
Tetrachloroguaiacol	96	95	109	104	100	102	120	104
Dihydrojuvabione	-	-	-	-	97	94	103	98
Juvabiol	-	-	-	-	96	94	100	92
Linoleic Acid	48	71	80	76	98	97	92	89
Linolenic Acid	60	95	102	100	104	98	81	79
Pimaric Acid	64	85	100	92	104	96	95	93
Epoxystearic Acid	77	84	95	105	105	98	108	107
Isopimaric Acid	62	87	94	94	102	95	94	95
Dehydroabietic Acid	75	92	105	96	110	100	113	103
Abietic Acid	59	64	84	76	93	74	(50)	(41)
Diepoxystearic Acid	77	84	(135)	(180)	-	-	-	-
Dichlorostearic Acid	70	73	87	107	93	91	95	86
Monochlorodehydroabietic Acid	76	88	92	108	110	105	111	115
Dichlorodehydroabietic Acid	72	85	84	105	106	104	101	96
Mean ± SD	70±11.8	85±10.2	94±9.5	99±13.3	101±5.6	96±7.5	101±10.2	98±10.7
Range	48-96	64-99	84-102	76-123	93-110	74-105	81-120	70-115

( ) outliers omitted from calculations of mean ± SD and range.

Toxicant solution: pH 7.0, specific conductance 5.0 mmho/cm.

Internal standard: heptadecanoic acid.

TABLE A5 RECOVERY EFFICIENCY USING PENTADECANOIC ACID AS INTERNAL STANDARD

Toxic Compound	% Recovery					
	1	2	3	4	5	6
Tetrachloroguaiacol	103	110	103	102	-	-
Dihydrojuvabione	116	113	108	115	110	81
Juvabione	111	106	101	108	102	81
Juvabiol	113	105	102	109	103	80
$\Delta 1'$-Dehydrojuvabione	-	-	-	-	107	80
Linoleic Acid	129	122	102	114	-	-
Linolenic Acid	123	108	92	116	-	-
Pimaric Acid	120	113	99	114	-	-
Epoxystearic Acid	126	136	106	121	-	-
Pimarol	-	-	-	-	60	64
Isopimaric Acid	123	112	103	121	-	-
Dehydroabietic Acid	120	118	108	125	-	-
Isopimarol	-	-	-	-	60	65
Abietic Acid	118	75	58	90	-	-
Dichlorostearic Acid	109	102	94	98	-	-
Chlorodehydroabietic Acid	120	117	109	128	-	-
Dichlorodehydroabietic Acid	112	111	90	109	-	-
Mean ± SD	117±6.6	111±13.3	98±13.0	112±10.4	-	-
Range	103-129	75-136	58-109	90-128	-	-

Toxicant solution: pH 7.0; specific conductance 5.0 mmho/cm.

TABLE A6 RECOVERY EFFICIENCY USING NONADECANOIC ACID AS INTERNAL STANDARD

Toxic Compound	% Recovery			
	1	2	3	4
Trichloroguaiacol	(161)	(142)	(145)	(134)
Tetrachloroguaiacol	124	107	105	101
Dihydrojuvabione	106	105	102	99
Juvabione	100	92	93	93
Juvabiol	82	90	91	88
Linoleic Acid	83	91	95	90
Linolenic Acid	63	81	86	78
Pimaric Acid	88	99	102	93
Epoxystearic Acid	99	103	106	95
Isopimaric Acid	80	91	94	90
Dehydroabietic Acid	98	103	102	100
Abietic Acid	(36)	(34)	(48)	(39)
Dichlorostearic Acid	102	98	87	84
Monochlorodehydroabietic Acid	101	103	98	97
Dichlorodehydroabietic Acid	100	100	89	91
Mean ± SD	94±15	97±7.6	96±6.8	92±6.5

( ) outliers omitted from calculations of mean ± SD and range

Toxicant solution: pH 7.0; specific conductance 5.1 mmho/cm

TABLE A7 VARIATION OF RESPONSE FACTORS AND RETENTION TIMES OF COMPOUNDS

Internal Standard	No. of Analyses	Trichloro-guaiacol	Tetrachloro-guaiacol	Oleic Acid	Linoleic Acid	Linolenic Acid	Pimaric Acid	Epoxy-stearic Acid	Iso-pimaric Acid	Dehydro-abietic Acid
Response Factor C_{17}	24	1.92±0.11	2.06±0.04	0.84±.08	1.01±0.04	-	1.05±0.04	0.97±.03	0.83±.02	0.87±0.04
C_{19}	13	1.85±0.12	1.91±0.08	-	1.12±0.08	1.02±0.09	1.13±0.04	1.10±.03	0.88±.05	0.98±0.03
Retention Time (min)	37	7.91±0.35	8.92±0.40	16.14±0.54	15.42±.82	14.91±0.45	22.64±1.15	23.56±1.21	25.45±1.31	27.04±1.44

Internal Standard	No. of Analyses	Abietic Acid	Diepoxy-stearic Acid	Dichloro-stearic Acid	Monochloro-dehydroabietic Acid	Dichloro-dehydroabietic Acid	Dihydro-Juvabione	Juvabione	Juvabiol
Response Factor C_{17}	24	1.47±.11	3.22±0.18	4.43±0.17	0.95±0.02	1.11±0.05	-	-	-
C_{19}	13	1.73±.21	-	4.82±0.17	1.05±0.03	1.35±0.18	1.48±0.05	1.25±0.05	2.87±0.06
Retention Time (min)	37	30.54±1.67	33.75±1.21	39.10±2.10	42.09±2.29 45.61±2.54	67.57±3.62	10.35±0.02 11.28±0.02	12.80±0.02	13.50±0.03 13.82±0.03

TABLE A8 COMPARISON OF RESULTS FOR DIRECT ETHER EXTRACTION AND RESIN ADSORPTION METHODS FOR A SAMPLE FROM MILL D

Toxicant	Concentration (mg/litre)	
	Direct ether extraction	XAD-2 adsorption
Trichloroguaiacol	0.25	0.12
Tetrachloroguaiacol	0.18	0.52
Juvabione	0.59	0.12
Linoleic Acid	0.20	0.35
Linolenic Oleic Acid	0.12	0.41
Pimaric Acid	0.33	0.61
Isopimaric Acid	0.41	0.58
Levopimaric Acid	0.10	0.43
Dehydroabietic Acid	0.75	1.65
Abietic Acid	0.30	0.92
Monochlorodehydroabietic Acid	0.55	0.65
Dichlorodehydroabietic Acid	0.39	0.59
TOTAL	4.17	6.95

TABLE A9 ADSORPTION EFFICIENCY OF XAD-2 RESIN DETERMINED BY SOLVENT EXTRACTION OF PERCOLATED BLEACHED KRAFT WHOLE MILL EFFLUENTS

Toxicants	Concentration (mg/litre)							
	Sample 1, Mill A				Sample 2, Mill D			
	Adsorbed by XAD-2 resin (Duplicates)		Percolate from XAD-2 resin (Duplicates)		Adsorbed by XAD-2 resin (Duplicates)		Percolate from XAD-2 resin (Duplicates)	
Trichloroguaiacol	0.10	0.10	0.004	0.005	0.21	0.24	0.005	0.014
Tetrachloroguaiacol	0.09	0.09	0.009	0.010	0.61	0.62	0.003	0.011
Juvabione	0.49	0.70	0.011	0.011	0.22	0.22	<0.001	0.004
Linoleic Acid	0.09	0.09	0.010	0.011	0.41	0.49	0.007	0.010
Linolenic Acid	0.17	0.19	0.006	0.006	0.46	0.56	0.004	0.007
Pimaric Acid	1.59	1.50	0.014	0.014	0.70	0.73	0.007	0.010
Isopimaric Acid	0.68	0.67	0.006	0.008	0.62	0.74	0.007	0.009
Levopimaric Acid	0.57	0.55	0.007	0.008	0.45	0.42	0.003	<0.001
Dehydroabietic Acid	5.26	4.90	0.024	0.026	1.76	1.74	0.012	0.014
Abietic Acid	1.53	1.54	0.018	0.020	1.01	1.02	0.013	0.016
Monochlorodehydroabietic Acid	0.09	0.09	0.007	0.008	0.71	0.79	0.015	0.015
Dichlorodehydroabietic Acid	0.08	0.08	0.017	0.020	0.63	0.75	0.016	0.023
TOTAL	10.74	10.50	0.133	0.147	7.79	8.32	0.092	0.133

TABLE A10 ADSORPTION EFFICIENCY OF XAD-2 RESIN DETERMINED BY PASSING EFFLUENT THROUGH TWO COLUMNS IN SERIES

Toxicant	Concentration (mg/litre)	
	1st Column XAD-2 Resin	2nd Column XAD-2 Resin
Trichloroguaiacol	ND	ND
Tetrachloroguaiacol	ND	ND
Juvabione	0.64	0.04
Linoleic Acid	ND	ND
Linolenic Acid	ND	0.07
Pimaric Acid	1.49	0.04
Isopimaric Acid	0.60	ND
Levopimaric Acid	0.30	ND
Dehydroabietic Acid	5.01	0.10
Abietic Acid	1.16	ND
Monochlorodehydroabietic Acid	ND	ND
Dichlorodehydroabietic Acid	ND	ND
TOTAL	9.20	0.25

ND = not detected (<0.03 mg/litre)

TABLE B1 CONCENTRATIONS OF TOXIC COMPOUNDS
IN PRIMARY-TREATED UNBLEACHED SOFTWOOD KRAFT EFFLUENTS, MILLS A,F,S,U,W

TOXICANT	SAMPLE	CONCENTRATION (mg/l)						
		A1 Duplicates		A2 Triplicates			F28	F29
Trichloroguaiacol		-	-	-	-	-	-	-
Tetrachloroguaiacol		-	-	-	-	-	-	-
Juvabione/Todomatuic Acid		0.31	0.25	0.39	0.60	0.64	0.16	0.43
Linoleic Acid		0.11	<0.05	<0.05	<0.05	<0.05	1.59	5.26
Oleic Acid		0.07	<0.05	0.07	0.09	<0.05	<0.05	<0.05
Pimaric/Sandaracopimaric Acid		1.17	1.32	1.49	1.40	1.49	0.50	1.92
Isopimaric Acid		0.57	0.66	0.58	0.56	0.60	0.89	3.58
Levopimaric Acid		0.34	0.38	0.47	0.45	0.30	0.87	4.72
Dehydroabietic Acid		3.86	4.55	5.16	4.80	5.01	1.50	5.31
Abietic Acid		1.51	1.72	1.53	1.54	1.16	1.82	7.05
Neoabietic Acid		<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
Total Toxic Units		9.5	10.9	11.4	11.1	10.7	8.1	32.9
Calculated 96-h LC50 (% by vol.)		10.5	9.2	8.8	9.0	9.3	12.3	3.0
Measured 96-h LC50 (% by vol.)		8.3			5.5		2.2	1.7
95% confidence limits		7.5-9.5			4.5-6.7		2.1-2.4	1.6-1.9

continued

NT - not toxic

TABLE B1(continued)

TOXICANT	SAMPLE	CONCENTRATION (mg/l) S1	U1	U2*	W2	W3*
Trichloroguaiacol		-	0.35	0.27	0.26	0.58
Tetrachloroguaiacol		-	<0.01	0.18	0.24	0.18
Juvabione/Todomatuic Acid		< 0.01	<0.01	<0.01	0.29	<0.01
Linoleic Acid		4.6	0.41	0.13	0.68	0.07
Oleic Acid		2.1	0.39	0.14	0.21	0.06
Pimaric/Sandaracopimaric Acid		1.4	0.07	<0.01	0.29	<0.01
Isopimaric Acid		1.7	0.88	0.12	0.78	0.03
Levopimaric Acid		2.7	0.37	<0.01	0.84	<0.01
Dehydroabietic Acid		4.7	2.20	0.76	0.57	0.05
Abietic Acid		5.6	0.88	0.08	0.82	0.02
Neoabietic Acid		1.2	<0.01	<0.01	0.31	<0.01
Total Toxic Units		23.8	5.4	<1	6.4	<1
Calculated 96-h LC50 (% by vol.)		4.2	18.5	NT	15.6	NT
Measured 96-h LC50 (% by vol.)		6.4	20.4	NT	10.9	NT
95% confidence limits		5.9-7.0	18.1-22.4	-	-	-

NT - not toxic

TABLE B2 CONCENTRATIONS OF TOXIC COMPOUNDS IN PRIMARY-TREATED BLEACHED SOFTWOOD KRAFT EFFLUENTS, MILLS B,C,N,Y

TOXICANT	SAMPLE	CONCENTRATION (mg/l)								
		B1 Duplicates		C1 Duplicates		N1	N2	N3	N4	Y1*
Trichloroguaiacol		0.05	0.06	0.22	0.23	<0.01	0.02	0.03	0.07	0.18
Tetrachloroguaiacol		<0.05	<0.05	0.62	0.60	<0.01	0.09	0.03	<0.01	0.30
Juvabione/Todomatuic Acid		<0.05	<0.05	0.08	0.07	<0.01	<0.01	0.04	<0.01	0.32
Linoleic Acid		0.71	0.74	0.73	0.73	<0.01	0.21	0.26	0.29	0.13
Oleic Acid		0.70	0.75	0.23	0.22	0.04	0.20	0.18	0.30	0.34
Pimaric/Sandaracopimaric Acid		0.49	0.47	0.38	0.39	<0.01	0.04	0.08	0.07	<0.01
Isopimaric Acid		1.16	1.17	0.49	0.55	0.07	0.18	0.29	0.27	0.97
Levopimaric Acid		<0.05	<0.05	<0.05	<0.05	<0.01	0.11	0.21	0.24	0.58
Dehydroabietic Acid		1.40	1.22	1.70	1.76	0.99	1.07	1.60	2.14	0.92
Abietic Acid		1.39	1.56	0.47	0.44	0.03	0.17	0.33	0.33	0.97
Monochlorodehydroabietic Acid		0.07	<0.05	0.75	0.75	<0.01	<0.01	<0.01	0.11	<0.01
Dichlorodehydroabietic Acid		<0.05	<0.05	0.41	0.38	<0.01	<0.01	<0.01	<0.01	<0.01
Total Toxic Units		7.5	7.6	8.8	9.0	1.0	1.8	2.7	3.2	6.8
Calculated 96-h LC50 (% by vol.)		13.3	13.2	11.4	11.1	100	55	33	31	14.7
Measured 96-h LC50 (% by vol.)		16.0		-		56	20	16.3	32	10.2
95% confidence limits		14.0-18.2		-		48.2-67.2	17.7-22.5	14.8-17.8	29.0-34.1	9.0-11.3

* Mixed softwoods and hardwoods

TABLE B3 CONCENTRATIONS OF TOXIC COMPOUNDS IN PRIMARY-TREATED BLEACHED SOFTWOOD KRAFT EFFLUENTS, MILL D

TOXICANT	SAMPLE	CONCENTRATION (mg/l)						
		D1 Duplicates		D2	D3	D4	D5	D6
Trichloroguaiacol		0.11	0.14	0.06	0.05	0.06	0.08	0.06
Tetrachloroguaiacol		0.52	0.52	<0.05	<0.05	<0.05	0.06	<0.05
Juvabione/Todomatuic Acid		0.12	0.12	0.14	0.12	0.08	0.05	0.14
Linoleic Acid		0.31	0.38	0.57	0.92	0.21	<0.05	0.58
Oleic Acid		0.36	0.46	<0.05	<0.05	<0.05	<0.05	<0.05
Pimaric/Sandaracopimaric Acid		0.60	0.62	0.43	0.62	0.22	0.15	0.58
Isopimaric Acid		0.52	0.64	0.72	1.10	0.35	0.19	0.97
Levopimaric Acid		<0.05	<0.05	0.58	0.92	0.27	0.16	0.74
Dehydroabietic Acid		1.66	1.64	2.20	2.90	1.00	0.74	2.40
Abietic Acid		0.91	0.92	1.60	2.20	0.82	0.49	2.10
Monochlorodehydroabietic Acid		0.61	0.69	0.09	0.80	0.09	0.06	0.10
Dichlorodehydroabietic Acid		0.53	0.65	0.06	<0.05	0.05	<0.05	0.05
Total Toxic Units		8.8	10.0	5.5	8.2	2.7	1.9	7.1
Calculated 96-h LC50 (% by vol.)		11.4	10.0	18.2	12.2	37	53	14.1
Measured 96-h LC50 (% by vol.)		15.7		13.7	14.9	18.1	45	17.3
95% confidence limits		12.1-20.5		13.0-14.4	14.0-15.8	17.4-19.2	42.1-47.5	16.0-18.7

TABLE B4 CONCENTRATIONS OF TOXIC COMPOUNDS
IN PRIMARY-TREATED BLEACHED SOFTWOOD KRAFT EFFLUENTS, MILL E

TOXICANT	SAMPLE	CONCENTRATION (mg/1) E1	E2	E3	E4	E5
Trichloroguaiacol		<0.05	0.05	0.07	0.08	0.08
Tetrachloroguaiacol		<0.05	<0.05	<0.05	<0.05	<0.05
Juvabione/Todomatuic Acid		0.19	0.17	0.19	0.14	0.15
Linoleic Acid		<0.05	0.12	0.30	0.05	<0.05
Pimaric/Sandaracopimaric Acid		0.26	0.41	0.40	0.28	0.32
Isopimaric Acid		0.45	0.80	0.78	0.53	0.64
Levopimaric Acid		0.36	0.68	0.74	0.56	0.72
Dehydroabietic Acid		0.96	1.45	1.37	1.02	1.06
Abietic Acid		1.08	1.69	1.52	1.11	1.38
Monochlorodehydroabietic Acid		0.29	0.15	0.18	0.17	0.20
Dichlorodehydroabietic Acid		0.12	<0.05	0.08	0.12	0.09
Total Toxic Units		4.4	6.7	6.6	4.8	5.8
Calculated 96-h LC50 (% by vol.)		22.7	14.9	15.2	20.8	17.2
Measured 96-h LC50 (% by vol.)		12.9	9.1	8.0	15.4	11.2
95% confidence limits		11.0-15.1	7.7-10.8	6.9-9.3	12.0-19.8	9.2-13.5

TABLE B5 CONCENTRATIONS OF TOXIC COMPOUNDS IN PRIMARY-TREATED BLEACHED SOFTWOOD KRAFT EFFLUENTS, MILL F

TOXICANT	SAMPLE	CONCENTRATION (mg/l)									
		F1	F2	F3	F4	F5	F6	F7	F8	F9	F10
Trichloroguaiacol		0.10	0.09	0.12	0.18	0.06	0.08	0.07	0.07	0.07	<0.05
Tetrachloroguaiacol		0.08	0.11	0.14	0.17	0.06	0.10	0.07	0.09	0.09	<0.05
Juvabione/Todomatuic Acid		0.20	0.17	0.33	0.22	0.29	0.17	0.32	0.24	0.17	0.61
Linoleic Acid		1.42	0.06	0.19	0.05	1.22	<0.05	0.60	<0.05	0.07	2.99
Pimaric/Sandaracopimaric Acid		0.45	0.52	0.53	0.47	0.36	0.50	0.56	0.45	0.41	0.81
[illegible]pimaric Acid		0.77	0.96	1.01	0.75	0.76	0.69	1.02	0.68	0.64	2.05
[illegible]vopimaric Acid		0.99	0.94	0.87	0.61	0.63	0.18	0.56	<0.05	0.09	1.48
Dehydroabietic Acid		1.31	1.66	1.77	1.73	1.22	1.72	1.90	1.76	1.45	4.46
Abietic Acid		1.55	1.90	1.85	1.76	1.27	1.10	1.96	0.29	0.93	4.72
Monochlorodehydroabietic Acid		0.15	0.26	0.31	0.30	0.24	0.27	0.28	0.30	<0.05	<0.05
Dichlorodehydroabietic Acid		<0.05	<0.05	<0.05	0.08	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
Total Toxic Units		7.7	8.5	9.0	8.1	7.0	6.0	8.7	5.0	4.5	19.0
Calculated 96-h LC50 (% by vol.)		13.0	11.8	11.1	12.3	14.3	16.7	11.5	20.0	22.2	5.3
Measured 96-h LC50 (% by vol.)		9.2	8.5	8.4	11.2	11.2	12.3	10.5	10.4	14.9	5.2
95% confidence limits		8.8-9.6	8.1-9.0	7.9-8.9	8.3-15.1	9.0-14.0	10.0-15.2	9.6-11.5	9.4-11.4	14.0-15.9	4.6-5.8

TABLE B6 CONCENTRATIONS OF TOXIC COMPOUNDS IN SECONDARY-TREATED BLEACHED SOFTWOOD KRAFT EFFLUENTS, MILLS D,N,Y

TOXICANT	SAMPLE	CONCENTRATION (mg/l)								
		D7	D8	D9	D10	D11	N5	N6	N7	Y2*
Trichloroguaiacol		0.05	0.06	0.07	0.06	0.06	<0.01	0.02	0.02	<0.01
Tetrachloroguaiacol		0.05	<0.05	0.05	0.06	<0.05	<0.01	0.06	0.01	0.06
Juvabione/Todomatuic Acid		<0.05	<0.05	<0.05	<0.05	<0.05	<0.01	<0.01	0.01	<0.01
Oleic Acid		<0.05	<0.05	<0.05	<0.05	<0.05	0.05	0.09	0.06	0.05
Linoleic Acid		<0.05	<0.05	<0.05	<0.05	<0.05	0.02	0.06	0.02	0.07
Pimaric/Sandaracopimaric Acid		<0.05	<0.05	<0.05	<0.05	<0.05	<0.01	0.02	0.01	<0.01
Isopimaric Acid		<0.05	<0.05	<0.05	<0.05	<0.05	0.04	0.02	0.02	<0.01
Levopimaric Acid		<0.05	<0.05	<0.05	<0.05	<0.05	<0.01	0.03	0.02	<0.01
Dehydroabietic Acid		0.07	0.21	<0.05	<0.05	<0.05	0.19	0.32	0.08	0.01
Abietic Acid		<0.05	0.13	<0.05	<0.05	<0.05	0.03	0.08	0.02	<0.01
Monochlorodehydroabietic Acid		<0.05	<0.05	<0.05	<0.05	<0.05	<0.01	<0.01	<0.01	<0.01
Dichlorodehydroabietic Acid		<0.05	<0.05	<0.05	<0.05	<0.05	<0.01	<0.01	<0.01	<0.01
Total Toxic Units		0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
Calculated 96-h LC50 (% by vol.)		NT	NT	NT	NT	NT	NT	NT	NT	NT
Measured 96-h LC50 (% by vol.)		NT	NT	NT	NT	NT	NT	NT	NT	NT

*Mixed softwoods and hardwoods

NT-not toxic

TABLE B7 CONCENTRATIONS OF TOXIC COMPOUNDS IN SECONDARY-TREATED BLEACHED SOFTWOOD KRAFT EFFLUENTS, MILL E

TOXICANT	SAMPLE	CONCENTRATION (mg/l) E6	E7	E8	E9	E10	E11	E12	E13	E14
Trichloroguaiacol		0.19	0.09	0.06	0.08	0.07	0.07	0.10	0.08	0.10
Tetrachloroguaiacol		0.18	0.10	0.12	0.08	0.12	0.07	0.10	0.08	0.16
Juvabione/Todomatuic Acid		0.11	0.08	0.10	0.11	0.09	0.07	0.14	0.16	0.19
Linoleic Acid		0.12	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	0.89	0.52
Pimaric/Sandaracopimaric Acid		0.07	<0.05	<0.05	<0.05	0.10	<0.05	0.10	0.47	0.35
Isopimaric Acid		0.06	<0.05	<0.05	<0.05	0.15	0.06	0.13	0.93	0.74
Levopimaric Acid		0.08	<0.05	<0.05	<0.05	0.13	0.07	0.15	1.19	0.86
Dehydroabietic Acid		0.09	<0.05	0.12	0.21	0.66	0.31	0.55	1.75	1.47
Abietic Acid		<0.05	<0.05	0.07	0.10	0.26	0.15	0.26	1.78	1.27
Monochlorodehydroabietic Acid		0.15	<0.05	<0.05	<0.05	0.11	0.07	0.12	0.20	0.08
Dichlorodehydroabietic Acid		0.10	<0.05	<0.05	<0.05	<0.05	<0.05	0.07	0.05	0.08
Total Toxic Units		1.1	0.4	0.5	0.5	1.9	0.6	1.7	7.6	5.8
Calculated 96-h LC50 (% by vol.)		90.9	NT	NT	NT	52.6	NT	58.8	13.2	17.2
Measured 96-h LC50 (% by vol.)		NT	NT	NT	NT	NT	NT	NT	13.1	16.3
95% confidence limits		-	-	-	-	-	-	-	12.0-14.2	15.3-17.4

NT - not toxic

continued

TABLE B7(continued)

TOXICANT	SAMPLE	CONCENTRATION (mg/l) E15	E16	E17	E18	E19	E20
Trichloroguaiacol		0.19	0.06	0.08	0.08	0.08	0.08
Tetrachloroguaiacol		0.22	<0.05	<0.05	<0.05	<0.05	<0.05
Juvabione/Todomatuic Acid		0.28	0.08	0.08	0.11	0.09	0.08
Linoleic Acid		0.26	<0.05	<0.05	<0.05	<0.05	<0.05
Pimaric/Sandaracopimaric Acid		0.27	0.14	0.13	0.26	0.12	0.10
Isopimaric Acid		0.56	0.22	0.21	0.33	0.19	0.16
Levopimaric Acid		0.72	0.14	0.16	0.22	0.14	0.10
Dehydroabietic Acid		1.19	1.39	1.56	2.14	1.40	1.40
Abietic Acid		0.98	0.50	0.47	0.80	0.46	0.41
Monochlorodehydroabietic Acid		0.15	0.18	0.19	0.26	0.18	0.17
Dichlorodehydroabietic Acid		0.12	<0.05	<0.05	0.06	<0.05	<0.05
Total Toxic Units		5.7	3.2	3.3	5.0	3.1	3.0
Calculated 96-h LC50 (% by vol.)		17.5	31.2	30.3	20.0	32.2	33.3
Measured 96-h LC50 (% by vol.)		16.6	14.2	16.4	15.1	14.6	42.5
95% confidence limits		13.9-19.9	12.5-16.0	13.8-19.4	13.3-17.2	12.9-16.6	28.6-63.3

TABLE B8 CONCENTRATIONS OF TOXIC COMPOUNDS IN SECONDARY-TREATED BLEACHED SOFTWOOD KRAFT EFFLUENTS, MILL F

TOXICANT	SAMPLE	CONCENTRATION (mg/l) F11	F12	F13	F14	F15	F16	F17	F18	F19
Trichloroguaiacol		<0.05	0.05	<0.05	<0.05	0.06	0.07	0.09	0.11	0.10
Tetrachloroguaiacol		<0.05	<0.05	<0.05	<0.05	0.06	0.08	0.11	0.12	0.11
Juvabione/Todomatuic Acid		<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
Pimaric/Sandaracopimaric Acid		<0.05	0.16	0.07	<0.05	0.13	<0.05	<0.05	<0.05	0.07
Isopimaric Acid		<0.05	0.15	<0.05	0.06	<0.13	<0.05	<0.05	<0.05	0.09
Levopimaric Acid		<0.05	0.24	0.06	0.22	0.17	0.09	0.05	0.08	0.06
Dehydroabietic Acid		<0.05	0.37	0.44	0.63	0.71	0.51	0.40	0.47	0.50
Abietic Acid		<0.05	0.39	0.27	0.30	0.44	0.24	0.18	0.30	0.29
Monochlorodehydroabietic Acid		<0.05	<0.05	<0.05	<0.05	<0.05	0.07	<0.05	<0.05	<0.05
Dichlorodehydroabietic Acid		<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
Total Toxic Units		<0.2	1.5	0.8	1.4	2.1	1.1	1.0	1.5	1.4
Calculated 96-h LC50 (% by vol.)		NT	66.7	NT	71.4	47.6	90.9	100	66.7	71.4
Measured 96-h LC50 (% by vol.)		NT	90.0	NT	27.4	NT	NT	NT	NT	57.4
95% confidence limits		-	84.5-97.8	26.1-28.8	-	-	-	-	-	56.1-58.8

continued

TABLE B8(continued)

TOXICANT / SAMPLE	CONCENTRATION (mg/l) F20	F21	F22	F23	F24	F25	F26	F27
Trichloroguaiacol	0.11	0.11	0.06	0.09	0.09	0.12	0.11	0.08
Tetrachloroguaiacol	0.12	0.12	0.08	0.12	0.10	0.12	0.13	0.10
Juvabione/Todomatuic Acid	<0.05	<0.05	<0.05	<0.05	<0.05	0.06	0.08	0.15
Pimaric/Sandaracopimaric Acid	0.08	<0.05	<0.05	<0.05	<0.05	0.05	0.07	0.13
Isopimaric Acid	0.09	<0.05	0.06	<0.05	0.10	0.09	0.13	0.20
Levopimaric Acid	0.09	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	0.11
Dehydroabietic Acid	0.57	0.19	0.19	<0.05	0.10	0.08	0.07	0.27
Abietic Acid	0.40	0.15	0.19	<0.05	0.13	0.10	0.17	0.38
Monochlorodehydroabietic Acid	<0.05	<0.05	<0.05	<0.05	<0.05	0.06	<0.05	0.07
Dichlorodehydroabietic Acid	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
Total Toxic Units	1.9	1.0	0.6	0.4	0.5	0.8	1.0	1.6
Calculated 96-h LC50 (% by vol.)	52.6	100	NT	NT	NT	NT	100	62.5
Measured 96-h LC50 (% by vol.)	57.4	NT	NT	92.7	89.4	89.4	94.9	64.8
95% confidence limits	56.1-58.8	-	-	85.9-99.0	84.2-95.1	84.2-95.1	92.2-97.6	62.1-67.6

TABLE B9 CONCENTRATIONS OF TOXIC COMPOUNDS IN HARDWOOD KRAFT EFFLUENTS, MILLS G,L,O,Y

TOXICANT	SAMPLE	CONCENTRATION (mg/l)				
		Primary-treated		Secondary-treated		
		L1	O1	G1	L2	O2
Trichloroguaiacol		<0.01	<0.01	0.02	0.01	0.04
Tetrachloroguaiacol		0.15	0.28	0.07	0.04	0.15
Juvabione/Todomatuic Acid		0.10	<0.01	0.10	0.02	<0.01
Oleic Acid		<0.01	0.43	0.04	0.02	0.20
Linoleic Acid		0.05	0.17	0.10	0.01	0.07
Pimaric/Sandaracopimaric Acid		0.05	<0.01	<0.01	<0.01	0.01
Isopimaric Acid		0.06	<0.01	<0.01	<0.01	0.02
Levopimaric Acid		0.07	<0.01	<0.01	<0.01	0.02
Dehydroabietic Acid		0.33	0.43	0.01	0.01	0.19
Abietic Acid		0.30	<0.01	<0.01	<0.01	0.05
Neoabietic Acid		<0.01	<0.01	<0.01	<0.01	0.02
Monochlorodehydroabietic Acid		<0.01	<0.01	<0.01	<0.01	0.09
Dichlorodehydroabietic Acid		<0.01	<0.01	<0.01	<0.01	0.02
Total Toxic Units		1.1	1.3	<0.2	<0.2	0.7
Calculated 96-h LC50 (% by vol.)		90	75	NT	NT	NT
Measured 96-h LC50 (% by vol.)		84	87	NT	NT	NT

TABLE B10 CONCENTRATIONS OF TOXIC COMPOUNDS IN UNBLEACHED AND BLEACHED PRIMARY-TREATED SULPHITE EFFLUENTS, MILLS H,I,J,K,O,P,Q,R,V,W,Z

TOXICANT / SAMPLE	CONCENTRATION (mg/l)									
	H1	I1	I2	J1	K1	O3*	P1	P2	Q1	R1
Trichloroguaiacol	<0.05	<0.05	<0.05	<0.05	<0.05	<0.01	0.05	0.03	<0.01	<0.01
Tetrachloroguaiacol	<0.05	<0.05	<0.05	<0.05	0.13	0.88	0.25	0.01	0.11	<0.01
Juvabione/Todomatuic Acid	1.36	0.16	0.21	0.23	0.47	0.27	0.08	0.03	0.02	0.11
Linoleic Acid	0.34	0.09	0.12	0.08	<0.05	0.99	0.09	0.05	0.06	0.08
Oleic Acid	<0.05	<0.05	<0.05	0.42	<0.05	1.18	0.10	0.04	0.12	0.12
Pimaric/Sandaracopimaric Acid	0.08	0.05	0.06	<0.05	<0.05	0.20	<0.01	<0.01	<0.01	<0.01
Isopimaric Acid	0.39	0.10	0.14	0.08	0.13	0.30	<0.01	0.01	0.02	0.03
Levopimaric Acid	0.51	0.16	0.10	<0.05	<0.05	<0.01	<0.01	<0.01	<0.01	<0.01
Dehydroabietic Acid	0.90	1.08	0.70	1.78	3.76	1.42	0.19	0.10	0.41	0.45
Abietic Acid	1.08	0.52	0.56	0.43	0.41	0.43	<0.01	0.01	0.05	0.06
Monochlorodehydroabietic Acid	<0.05	<0.05	<0.05	0.31	0.90	0.49	0.49	0.26	<0.01	<0.01
Dichlorodehydroabietic Acid	<0.05	<0.05	<0.05	<0.05	<0.05	0.25	0.04	<0.01	<0.01	<0.01
Total Toxic Units	3.8	1.9	1.4	3.4	5.5	6.6	1.4	0.4	0.5	0.5
Calculated 96-h LC50 (% by vol.)	26.3	52.6	71.4	29.4	18.2	15	71	NT	NT	NT
Measured 96-h LC50 (% by vol.)	7.9	54.8	95	50	9	19	32.4	97	>60	76
95% confidence limits	-	-	-	-	-	-	-	-	-	-

* Neutral sulphite semi-chemical

continued

TABLE B10(continued)

TOXICANT / SAMPLE	CONCENTRATION (mg/l) V1	W1†	Z1	Z2	Z3
Trichloroguaiacol	0.02	<0.01	0.19	0.04	0.06
Tetrachloroguaiacol	<0.01	0.30	0.25	0.18	0.19
Juvabione/Todomatuic Acid	0.63	<0.01	<0.01	<0.01	0.04
Linoleic Acid	0.13	1.50	0.14	0.06	0.18
Oleic Acid	0.68	0.63	0.20	0.12	0.11
Pimaric/Sandaracopimaric Acid	0.40	<0.01	<0.01	<0.01	0.02
Isopimaric Acid	0.86	0.11	0.04	0.17	0.08
Levopimaric Acid	0.62	0.09	<0.01	<0.01	0.03
Dehydroabietic Acid	1.50	0.26	0.41	0.59	0.33
Abietic Acid	0.93	0.12	0.01	0.21	0.09
Monochlorodehydroabietic Acid	<0.01	<0.01	<0.01	<0.01	0.21
Dichlorodehydroabietic Acid	<0.01	<0.01	<0.01	<0.01	<0.01
Total Toxic Units	6.1	1.9	1.4	1.5	1.3
Calculated 96-h LC50 (% by vol.)	16.4	53	71	65	76
Measured 96-h LC50 (% by vol.)	32.2	47.6	15.7	25	65
95% confidence limits	30.3-34.6	42.8-52.4	12.8-18.0	22.6-26.8	-

* Neutral sulphite semi-chemical

TABLE B11 CONCENTRATIONS OF TOXIC COMPOUNDS IN SECONDARY-TREATED SULPHITE EFFLUENTS, MILLS K,P,Q,V,Z

TOXICANT	SAMPLE	CONCENTRATION (mg/1) K2	P3	P4	Q2	V2	Z4
Trichloroguaiacol		<0.05	0.06	0.01	<0.01	0.06	0.02
Tetrachloroguaiacol		<0.05	0.04	0.03	0.07	0.05	0.08
Juvabione/Todomatuic Acid		0.08	0.10	<0.01	<0.01	<0.01	<0.01
Oleic Acid		0.13	0.03	0.01	0.05	0.07	0.03
Linoleic Acid		0.05	0.04	<0.01	0.01	0.06	0.02
Isopimaric Acid		0.31	<0.01	<0.01	<0.01	0.03	0.01
Levopimaric Acid		0.06	<0.01	<0.01	<0.01	<0.01	0.01
Dehydroabietic Acid		0.70	0.15	0.01	0.08	0.09	0.02
Abietic Acid		0.09	<0.01	<0.01	<0.01	0.03	<0.01
Monochlorodehydroabietic Acid		0.09	0.45	<0.01	<0.01	<0.01	<0.01
Dichlorodehydroabietic Acid		<0.05	0.03	<0.01	<0.01	<0.01	<0.01
Total Toxic Units		1.4	0.7	<0.2	<0.2	<0.2	<0.2
Calculated 96-h LC50 (% by vol.)		71	NT	NT	NT	NT	NT
Measured 96-h LC50 (% by vol.)		45	80	NT	NT	NT	NT

NT - not toxic

TABLE B12 CONCENTRATIONS OF TOXIC COMPOUNDS IN GROUNDWOOD EFFLUENTS, MILLS M,T,X

TOXICANT	SAMPLE	CONCENTRATION (mg/l)									
		Primary-treated						Secondary-treated			
		M1	M2	M3	T1	X1	X2	M4	M5	T2	X3
Juvabione/Todomatuic Acid		1.4	1.7	1.6	0.20	1.20	0.38	1.6	1.3	<0.01	<0.01
Oleic Acid		3.0	4.3	4.2	0.23	0.41	0.38	0.9	1.4	0.05	0.09
Linoleic Acid		5.7	6.6	6.4	0.58	0.60	0.49	1.4	1.5	0.08	0.08
Pimaric/Sandaracopimaric Acid		5.7	6.8	4.8	0.02	0.34	0.10	4.6	5.7	0.02	0.03
Isopimaric Acid		8.3	9.3	7.0	0.15	0.49	0.29	6.5	7.9	0.09	0.06
Levopimaric Acid		19.8	22.0	16.7	0.08	0.64	0.30	1.8	0.9	0.02	0.02
Dehydroabietic Acid		15.1	12.5	11.0	0.49	1.20	0.58	0.7	0.9	0.11	0.05
Abietic Acid		12.6	15.8	11.3	0.21	0.77	0.47	4.1	0.5	0.07	0.07
Neoabietic Acid		6.2	6.8	5.2	0.03	0.16	0.09	3.8	3.4	<0.01	<0.01
Total Toxic Units		95.2	104.8	80.5	1.3	5.4	2.8	32.1	30.1	<0.2	<0.2
Calculated 96-h LC50 (% by vol.)		1.1	0.96	1.2	77	18.5	36	3.1	3.3	NT	NT
Measured 96-h LC50 (% by vol.)		1.2	1.1	1.3	60	55.4	100	3.5	3.9	NT	NT
95% confidence limits		1.11-1.42	1.02-1.22	1.23-1.42	55.7-64.0	50.6-59.7	-	3.05-4.08	3.61-4.23	-	-

TABLE C1 RECOVERIES (%) OF COMPOUNDS ADDED TO EFFLUENT SAMPLES

Compound	Sample															
	E12	E15	E20	D2	F27	L1	L2	M1	M2	M3	M4	M5	N2	N3	N4	N5
Trichloroguaiacol	120	102	77	136	112	90	98	100	68	102	90	110	110	106	74	104
Tetrachloroguaiacol	120	86	73	70	126	90	102	72	44	72	64	78	102	100	24	82
Juvabione/Todomatuic Acid	99	98	100	93	83	96	108	98	114	100	82	80	124	110	NA	114
Oleic Acid	NA	NA	NA	NA	NA	NA	NA	80	98	86	74	92	86	58	78	78
Linoleic Acid	93	76	67	58	56	80	104	80	132	84	86	96	98	62	68	92
Pimaric Acid	100	94	89	81	88	84	86	90	78	101	80	99	105	101	90	99
Isopimaric Acid	105	105	90	90	91	88	88	88	82	102	84	90	104	100	106	96
Levopimaric Acid	114	109	75	NA	94	70	62	70	68	82	76	102	120	120	74	84
Dehydroabietic Acid	91	103	49	39	95	84	82	64	76	82	94	104	96	78	84	86
Abietic Acid	96	103	103	74	65	79	86	91	84	99	90	114	102	99	102	88
Neoabietic Acid	NA	NA	NA	NA	NA	NA	NA	91	85	98	89	104	104	103	NA	80
Monochlorodehydroabietic Acid	94	82	84	91	75	108	113	99	100	100	85	109	101	94	101	91
Dichlorodehydroabietic Acid	77	57	69	60	46	112	107	98	95	111	86	113	93	83	95	90

NA - not added

continued

TABLE C1(continued)

Compound	Sample															
	N6	N7	O1	O2	O3	P1	P2	Q1	R1	S1	T1	U1	U2	V1	W1	W2
Trichloroguaiacol	104	94	94	94	104	118	98	112	92	90	106	102	110	118	108	92
Tetrachloroguaiacol	74	78	88	98	84	80	102	84	78	66	76	104	56	98	72	84
Juvabione/Todomatuic Acid	90	106	96	96	92	100	92	90	94	120	92	102	86	82	98	92
Oleic Acid	86	84	70	64	96	44	64	72	84	30	64	84	80	60	98	100
Linoleic Acid	98	98	84	84	110	68	80	90	94	34	86	94	86	70	104	76
Pimaric Acid	124	101	90	92	97	97	88	105	97	108	97	99	101	101	99	90
Isopimaric Acid	114	100	88	90	98	96	86	104	98	114	92	92	96	118	96	104
Levopimaric Acid	112	110	82	60	94	82	60	72	84	102	88	78	78	82	80	96
Dehydroabietic Acid	104	90	84	88	102	104	92	122	92	120	96	126	92	124	102	94
Abietic Acid	112	102	90	91	100	94	80	99	95	120	94	95	101	105	95	94
Neoabietic Acid	107	105	94	94	100	86	64	81	86	95	89	95	95	80	86	89
Monochlorodehydroabietic Acid	109	93	87	87	96	92	94	106	95	106	92	103	101	92	96	99
Dichlorodehydroabietic Acid	102	94	77	74	93	64	89	98	95	96	70	94	101	88	94	84

continued

TABLE C1(continued)

Compound	Sample					
	X1	X2	Y1	Z1	Z2	Z3
Trichloroguaiacol	108	100	118	104	114	102
Tetrachloroguaiacol	86	90	104	100	102	92
Juvabione/Todomatuic Acid	62	78	86	94	96	92
Oleic Acid	70	84	86	92	84	62
Linoleic Acid	94	94	96	98	94	78
Pimaric Acid	99	97	101	97	101	90
Isopimaric Acid	100	94	104	96	102	94
Levopimaric Acid	96	92	66	68	84	84
Dehydroabietic Acid	112	96	114	100	110	92
Abietic Acid	101	93	101	92	98	92
Neoabietic Acid	88	87	87	72	84	87
Monochlorodehydroabietic Acid	85	95	102	98	105	97
Dichlorodehydroabietic Acid	62	74	102	99	100	79

TABLE C2 MEAN RECOVERIES (± STANDARD DEVIATIONS) OF COMPOUNDS ADDED TO EFFLUENT SAMPLES

Compound	Recovery (%) SD		Outliers†
	All Values	Adjusted for Outliers*	
Trichloroguaiacol	102.1 ± 13.1	103.7 ± 8.7	136,68,74,77
Tetrachloroguaiacol	84.2 ± 19.4	85.8 ± 12.3	126,120,56,44,24
Juvabione/Todomatuic Acid	95.3 ± 12.2	93.5 ± 8.0	124,120,114,62
Oleic Acid	77.0 ± 15.9	79.8 ± 12.1	44,30
Linoleic Acid	85.3 ± 17.3	87.1 ± 11.8	132,58,56,34
Pimaric/Sandaracopimaric Acid	95.7 ± 8.6	95.4 ± 6.8	124,78
Isopimaric Acid	96.9 ± 8.6	95.3 ± 6.8	118, 114
Levopimaric Acid	85.6 ± 16.7	82.8 ± 14.1	120,114
Dehydroabietic Acid	93.8 ± 18.1	96.6 ± 12.3	126,64,49,39
Abietic Acid	95.2 ± 10.4	96.0 ± 5.5	120,114,112,80,79,74,65
Neoabietic Acid	90.2 ± 9.8	91.8 ± 7.9	72,64
Monochlorodehydroabietic Acid	96.2 ± 8.3	96.4 ± 7.2	113,75
Dichlorodehydroabietic Acid	87.4 ± 16.2	89.4 ± 14.0	57,46

* Values greater than ± 2SD from original mean removed. Mean recalculated, and process repeated once more.

† Values removed to obtain revised mean.

TABLE D1 CONCENTRATIONS OF POTENTIALLY TOXIC CONSTITUENTS IN INFLUENTS AND EFFLUENTS, OCTOBER 15-19, 1979 AND INFLUENT SAMPLE COLLECTED DECEMBER 10, 1979, MILL 1

Compound	Date	Influent (mg/litre)						Effluent (mg/litre)				
		15.10.79	16.10.79	17.10.79	18.10.79	19.10.79	10.12.79	15.10.79	16.10.79	17.10.79	18.10.79	19.10.79
Juvabione/ Todomatuic Acid	diss.	1.2	1.2	1.0	0.78	1.0	0.20	<0.001	<0.001	0.006	<0.001	<0.001
	total	0.71	0.45	0.37	0.67	0.55	0.25	<0.001	<0.001	<0.001	0.009	<0.001
Linoleic Acid	diss.	0.13	0.15	0.17	0.06	0.15	0.23	0.007	0.016	0.012	0.014	0.010
	total	1.00	0.44	0.93	0.95	0.67	0.33	0.11	0.074	0.074	0.065	0.12
Oleic Acid	diss.	0.13	0.17	0.19	0.11	0.17	0.17	0.018	0.024	0.022	0.021	0.021
	total	0.53	0.19	0.50	0.46	0.39	0.21	0.047	0.031	0.031	0.028	0.056
Pimaric Acid/ Sandaracopimaric Acid	diss.	0.16	0.12	0.17	0.16	0.17	0.15	<0.001	<0.001	<0.001	<0.001	<0.001
	total	0.31	0.22	0.30	0.23	0.29	0.25	0.002	0.001	<0.001	0.001	0.002
Isopimaric Acid	diss.	0.60	0.43	0.57	0.53	0.61	0.86	<0.001	0.001	0.001	0.001	0.005
	total	1.3	0.86	1.0	1.1	1.1	1.4	0.040	0.035	0.038	0.028	0.065
Levopimaric Acid	diss.	0.87	0.58	0.83	0.76	0.82	0.84	<0.001	<0.001	<0.001	<0.001	<0.001
	total	1.4	0.90	1.1	1.0	1.1	1.4	0.011	0.013	0.015	0.011	0.032
Dehydroabietic Acid	diss.	0.98	0.70	0.95	0.98	0.88	1.1	<0.001	<0.001	<0.001	<0.001	<0.001
	total	1.3	0.96	1.1	1.2	1.1	1.6	0.032	0.031	0.036	0.026	0.061
Abietic Acid	diss.	0.76	0.53	0.71	0.64	0.78	0.81	<0.001	<0.001	<0.001	<0.001	0.004
	total	1.5	0.93	1.1	1.1	1.3	1.4	0.031	0.034	0.037	0.028	0.069
Neoabietic Acid	diss.	0.18	0.11	0.17	0.15	0.19	0.09	<0.001	<0.001	<0.001	<0.001	<0.001
	total	0.26	0.16	0.18	0.17	0.24	0.17	<0.001	<0.001	0.002	<0.001	0.009

TABLE D2 CONCENTRATIONS OF POTENTIALLY TOXIC CONSTITUENTS IN INFLUENTS AND EFFLUENTS, OCTOBER 15-19, 1979 AND INFLUENT SAMPLE COLLECTED DECEMBER 10, 1979, MILL 2

Compound	Date	Influent (mg/litre)						Effluent (mg/litre)				
		15.10.79	16.10.79	17.10.79	18.10.79	19.10.79	10.12.79	15.10.79	16.10.79	17.10.79	18.10.79	19.10.79
Juvabione/ Todomatuic Acid	diss.	<0.01	0.85	0.033	0.012	<0.01	<0.01	<0.001	0.002	0.017	<0.001	<0.001
	total	<0.01	<0.01	<0.01	<0.001	0.048	<0.01	0.019	0.014	0.013	0.014	0.011
Linoleic Acid	diss.	<0.01	0.082	0.039	0.036	0.036	0.034	0.007	0.008	0.011	0.013	0.012
	total	0.080	0.096	0.12	0.17	0.13	0.090	0.072	0.071	0.081	0.10	0.083
Oleic Acid	diss.	0.052	0.091	0.095	0.091	0.075	0.032	0.022	0.015	0.026	0.024	0.023
	total	0.052	0.085	0.093	0.072	0.085	0.053	*	*	*	*	*
Isopimaric Acid	diss.	<0.01	0.11	0.011	0.021	0.025	0.008	<0.001	<0.001	0.002	0.001	0.001
	total	0.018	0.032	0.039	0.034	0.044	0.035	0.008	0.010	0.010	0.011	0.010
Levopimaric Acid	diss.	<0.01	0.10	0.009	0.025	0.024	0.011	<0.001	<0.001	<0.001	<0.001	<0.001
	total	0.025	0.041	0.048	0.051	0.057	0.050	<0.001	<0.001	<0.001	<0.001	<0.001
Dehydroabietic Acid	diss.	<0.01	0.093	0.034	0.049	0.089	0.028	<0.001	0.002	0.004	<0.001	<0.001
	total	0.068	0.11	0.18	0.13	0.15	0.17	0.019	0.023	0.025	0.027	0.025
Abietic Acid	diss.	<0.01	0.11	0.015	0.025	0.035	<0.01	<0.001	<0.001	<0.001	<0.001	<0.001
	total	0.041	0.052	0.081	0.075	0.088	0.060	0.007	0.008	0.008	0.008	0.010

* Not measured - interference from unknown constituent

TABLE D3 CONCENTRATIONS OF POTENTIALLY TOXIC CONSTITUENTS IN INFLUENTS AND EFFLUENTS, OCTOBER 15-19, 1979 AND INFLUENT SAMPLE COLLECTED DECEMBER 11, 1979, MILL 3

Compound	Date	Influent (mg/litre)						Effluent (mg/litre)				
		15.10.79	16.10.79	17.10.79	18.10.79	19.10.79	11.12.79	15.10.79	16.10.79	17.10.79	18.10.79	19.10.79
Juvabione/ Todomatuic Acid	diss.	0.29	0.27	0.36	0.41	0.19	0.08	0.004	0.063	0.034	0.005	<0.001
	total	0.06	0.19	0.10	0.21	0.16	0.23	0.010	ND	ND	ND	ND
Linoleic Acid	diss.	0.11	0.07	0.21	0.04	0.15	0.08	0.016	0.013	0.019	0.012	0.008
	total	0.22	0.43	0.56	0.37	0.57	0.28	0.11	0.11	0.073	0.18	0.20
Oleic Acid	diss.	0.11	0.10	0.14	0.07	0.09	0.09	0.020	0.020	0.028	0.022	0.026
	total	0.12	0.27	0.30	0.23	0.39	0.15	0.042	0.062	0.058	0.076	0.086
Pimaric Acid/ Sandaracopimaric Acid	diss.	0.19	0.05	0.27	0.39	0.33	0.07	<0.001	<0.001	0.001	<0.001	<0.001
	total	0.10	0.21	0.34	0.38	0.35	0.17	0.005	0.030	0.006	0.050	0.057
Isopimaric Acid	diss.	0.57	0.19	0.79	1.2	1.1	0.21	0.002	0.001	0.001	<0.001	<0.001
	total	0.33	0.75	1.0	1.3	1.2	0.60	0.066	0.089	0.071	0.075	0.087
Levopimaric Acid	diss.	0.53	0.21	0.68	0.96	0.89	0.25	<0.001	<0.001	<0.001	<0.001	<0.001
	total	0.32	0.70	0.91	1.1	1.1	0.73	0.018	0.013	0.012	0.023	0.032
Dehydroabietic Acid	diss.	0.98	0.88	1.2	1.8	1.3	0.83	<0.001	<0.001	<0.001	<0.001	0.001
	total	0.41	0.98	1.5	1.4	1.4	0.87	0.037	0.075	0.060	0.065	0.076
Abietic Acid	diss.	0.69	0.21	0.87	1.2	1.2	0.24	<0.001	<0.001	<0.001	<0.001	<0.001
	total	0.32	0.58	0.94	1.1	1.1	0.57	0.033	0.045	0.039	0.043	0.061
Neoabietic Acid	diss.	0.15	0.06	0.26	0.35	0.33	0.06	<0.001	<0.001	<0.001	<0.001	<0.001
	total	0.06	0.10	0.19	0.22	0.25	0.18	<0.001	<0.001	<0.001	0.002	0.005

ND - not determined, interference from unidentified compound

TABLE D4 CONCENTRATIONS OF POTENTIALLY TOXIC CONSTITUENTS IN INFLUENTS AND EFFLUENTS, OCTOBER 15-19, 1979 AND INFLUENT SAMPLE COLLECTED DECEMBER 11, 1979, MILL 4

Compound	Date	Influent (mg/litre)						Effluent (mg/litre)				
		15.10.79	16.10.79	17.10.79	18.10.79	19.10.79	11.12.79	15.10.79	16.10.79	17.10.79	18.10.79	19.10.79
Juvabione/ Todomatuic Acid	diss.	0.19	0.22	0.42	0.39	0.23	0.04	0.008	<0.001	0.013	0.007	0.005
	total	0.10	0.18	0.27	0.26	0.25	0.09	<0.001	<0.001	<0.001	<0.001	0.008
Linoleic Acid	diss.	0.11	0.12	0.12	0.12	0.12	0.20	0.011	0.020	0.018	0.014	0.015
	total	0.41	0.52	0.57	0.45	0.51	0.30	0.18	0.35	0.22	0.19	0.23
Oleic Acid	diss.	0.12	0.09	0.10	0.13	0.13	0.14	0.024	0.024	0.026	0.015	0.018
	total	0.33	0.33	0.36	0.29	0.33	0.16	0.060	0.071	0.11	0.032	0.030
Pimaric Acid/ Sandaracopimaric Acid	diss.	<0.01	0.07	0.11	0.13	0.04	0.13	<0.001	<0.001	<0.001	<0.001	<0.001
	total	0.17	0.27	0.34	0.26	0.29	0.24	0.025	0.022	0.021	0.032	0.030
Isopimaric Acid	diss.	0.26	0.47	0.74	0.85	0.38	0.79	0.002	0.005	0.004	0.003	0.005
	total	1.3	2.0	2.4	2.1	1.9	1.5	0.35	0.31	0.53	0.44	0.49
Levopimaric Acid	diss.	0.12	0.31	0.53	0.69	0.35	0.33	<0.001	0.004	0.002	0.001	<0.001
	total	0.50	1.1	1.5	1.3	0.95	0.65	0.088	0.070	0.12	0.10	0.14
Dehydroabietic Acid	diss.	0.72	1.0	1.6	1.6	0.99	1.4	<0.001	0.004	0.004	0.004	<0.001
	total	1.3	2.0	2.2	1.6	1.9	2.1	0.21	0.31	0.34	0.29	0.28
Abietic Acid	diss.	0.29	0.54	0.88	1.0	0.45	0.9	<0.001	<0.001	<0.001	<0.001	0.004
	total	1.3	2.0	2.3	2.0	2.0	1.6	0.32	0.24	0.50	0.42	0.51
Neoabietic Acid	diss.	<0.01	0.12	0.28	0.35	0.19	0.12	<0.001	<0.001	<0.001	<0.001	<0.001
	total	0.12	0.25	0.40	0.38	0.33	0.21	0.020	<0.001	0.021	0.022	0.041

TABLE D5 CONCENTRATIONS OF POTENTIALLY TOXIC CONSTITUENTS IN INFLUENTS AND EFFLUENTS, OCTOBER 15-19, 1979 AND INFLUENT SAMPLE COLLECTED DECEMBER 10, 1979, MILL 5

Compound	Date	Influent (mg/litre)						Effluent (mg/litre)				
		15.10.79	16.10.79	17.10.79	18.10.79	19.10.79	10.12.79	15.10.79	16.10.79	17.10.79	18.10.79	19.10.79
Juvabione/ Todomatuic Acid	diss.	0.03	0.07	0.08	0.06	0.05	0.04	0.004	0.011	<0.001	<0.001	<0.001
	total	0.06	0.01	0.02	0.12	0.02	0.05	<0.001	<0.001	<0.001	<0.001	<0.001
Linoleic Acid	diss.	<0.01	0.04	0.04	0.04	0.06	0.07	0.009	0.013	0.010	0.014	0.014
	total	0.43	0.40	0.42	0.42	0.38	0.28	0.069	0.090	0.083	0.074	0.077
Oleic Acid	diss.	0.05	0.05	0.06	0.10	0.14	0.07	0.022	0.021	0.031	0.022	0.027
	total	0.20	0.13	0.13	0.15	0.14	0.11	0.028	0.041	0.047	<0.001	<0.001
Isopimaric Acid	diss.	<0.01	0.03	0.07	0.02	0.05	0.08	<0.001	0.004	0.002	<0.001	<0.001
	total	0.05	0.04	0.04	0.03	0.03	0.09	0.005	0.005	0.006	0.004	0.005
Levopimaric Acid	diss.	<0.01	<0.01	0.042	<0.01	0.04	0.03	<0.001	<0.001	<0.001	<0.001	<0.001
	total	<0.01	<0.01	<0.01	<0.01	<0.01	0.04	<0.001	<0.001	<0.001	<0.001	<0.001
Dehydroabietic Acid	diss.	0.24	0.28	0.34	0.33	0.43	0.80	0.002	<0.001	0.001	<0.001	<0.001
	total	0.35	0.39	0.31	0.27	0.34	0.81	0.020	0.023	0.020	0.019	0.017
Abietic Acid	diss.	<0.01	0.04	0.07	0.04	0.07	0.11	<0.001	<0.001	<0.001	<0.001	<0.001
	total	0.07	0.05	0.04	0.03	0.04	0.15	0.005	0.006	0.007	0.006	0.008

TABLE D6 CONCENTRATIONS OF POTENTIALLY TOXIC CONSTITUENTS IN INFLUENTS AND EFFLUENTS, OCTOBER 15-19, 1979 AND INFLUENT SAMPLE COLLECTED DECEMBER 11, 1979, MILL 6

Compound	Date	Influent (mg/litre)						Effluent (mg/litre)				
		15.10.79	16.10.79	17.10.79	18.10.79	19.10.79	11.12.79	15.10.79	16.10.79	17.10.79	18.10.79	19.10.79
Trichloroguaiacol	diss.	0.072	0.032	0.019	0.048	<0.01	0.051	0.026	0.094	0.024	0.021	0.020
	total	0.014	0.083	0.095	0.045	0.040	0.049	0.036	0.024	0.021	0.023	0.026
Tetrachloro-guaiacol	diss.	0.043	0.031	0.015	0.083	<0.01	0.059	0.068	0.19	0.060	0.052	0.034
	total	0.038	0.088	0.014	0.068	0.067	0.084	0.013	0.009	0.014	0.015	0.014
Juvabione/ Todomatuic Acid	diss.	0.014	0.082	0.28	0.11	0.13	0.033	<0.001	<0.001	0.010	<0.001	0.029
	total	0.078	0.035	0.034	0.064	0.042	0.068	<0.001	<0.001	<0.001	<0.001	0.004
Linoleic Acid	diss.	<0.01	0.042	0.056	0.044	0.046	0.025	0.007	0.009	0.009	0.014	0.008
	total	0.11	0.067	0.099	0.096	0.11	0.11	0.025	0.019	0.017	0.020	0.030
Oleic Acid	diss.	0.034	0.066	0.073	0.077	0.070	0.042	0.013	0.020	0.022	0.026	0.015
	total	0.045	0.022	0.048	0.034	0.034	0.042	0.011	0.012	0.009	<0.001	<0.001
Isopimaric Acid	diss.	0.043	0.056	0.095	0.045	0.057	0.21	<0.001	<0.001	<0.001	<0.001	0.003
	total	0.067	0.10	0.11	0.11	0.099	0.18	0.002	0.002	<0.001	<0.001	<0.001
Levopimaric Acid	diss.	<0.01	<0.01	0.033	0.008	0.003	0.046	<0.001	<0.001	<0.001	<0.001	<0.001
	total	0.092	0.075	0.092	0.077	0.075	0.089	<0.001	<0.001	<0.001	<0.001	<0.001
Dehydroabietic Acid	diss.	0.10	0.065	0.093	0.044	0.034	0.14	0.002	<0.001	<0.001	<0.001	0.006
	total	0.084	0.085	0.083	0.071	0.078	0.14	<0.001	<0.001	<0.001	<0.001	<0.001
Abietic Acid	diss.	<0.01	<0.01	0.032	0.014	<0.01	0.071	<0.001	<0.001	<0.001	<0.001	0.002
	total	0.036	0.019	0.018	0.020	0.013	0.084	<0.001	<0.001	<0.001	<0.001	<0.001

TABLE D7 CONCENTRATIONS OF POTENTIALLY TOXIC CONSTITUENTS IN INFLUENTS AND EFFLUENTS, OCTOBER 15-19, 1979 AND INFLUENT SAMPLE COLLECTED DECEMBER 10, 1979 MILL 7

Compound	Date	Influent (mg/litre)					
		15.10.79	16.10.79	17.10.79	18.10.79	19.10.79	10.12.79
Juvabione/ Todomatuic Acid	diss.	1.6	1.5	1.0	1.2	1.3	<0.01
	total	0.30	0.35	0.24	0.24	0.34	<0.01
Linoleic Acid	diss.	0.55	0.24	0.26	0.06	0.05	0.91
	total	1.5	1.8	1.5	1.2	0.83	1.2
Oleic Acid	diss.	0.23	0.19	0.18	0.09	0.07	0.52
	total	0.46	0.78	0.58	0.47	0.31	0.84
Pimaric Acid/ Sandaracopimaric Acid	diss.	<0.01	<0.01	0.11	<0.01	0.18	0.34
	total	0.10	0.16	0.21	0.11	0.08	0.76
Isopimaric Acid	diss.	0.69	0.78	1.4	0.40	1.2	1.8
	total	1.2	1.7	1.8	1.1	1.1	2.9
Levopimaric Acid	diss.	0.71	0.84	1.4	0.53	1.1	1.8
	total	1.2	1.4	1.6	0.96	1.0	3.1
Dehydroabietic Acid	diss.	0.73	0.70	1.2	0.68	1.3	1.7
	total	1.1	1.6	1.8	0.94	1.0	2.7
Abietic Acid	diss.	0.65	0.80	1.3	0.44	1.1	1.9
	total	1.2	1.5	1.5	1.0	1.1	3.2
Neoabietic Acid	diss.	0.95	0.45	0.31	0.14	0.14	0.36
	total	0.47	0.43	0.24	0.15	0.19	0.60

continued

TABLE D7(continued)

Compound	Date	Effluent (mg/litre)				
		15.10.79	16.10.79	17.10.79	18.10.79	19.10.79
Juvabione/ Todomatuic Acid	diss.	0.004	<0.001	<0.001	<0.001	<0.001
	total	<0.001	<0.001	<0.001	<0.001	<0.001
Linoleic Acid	diss.	0.013	0.001	0.009	0.009	0.010
	total	0.33	0.29	0.42	0.31	0.32
Oleic Acid	diss.	0.024	<0.001	0.021	0.021	0.022
	total	<0.001	0.09	0.13	0.12	0.09
Pimaric Acid/ Sandaracopimaric Acid	diss.	<0.001	<0.001	<0.001	0.001	0.002
	total	0.002	<0.001	<0.001	<0.001	0.001
Isopimaric Acid	diss.	0.002	<0.001	<0.001	0.006	0.002
	total	0.05	0.023	0.041	0.040	0.033
Levopimaric Acid	diss.	<0.001	<0.001	<0.001	<0.001	<0.001
	total	0.007	0.004	0.007	0.008	0.012
Dehydroabietic Acid	diss.	0.011	<0.001	0.005	0.010	0.008
	total	0.031	0.017	0.024	0.026	0.026
Abietic Acid	diss.	0.008	<0.001	0.003	0.003	0.011
	total	0.030	0.015	0.015	0.026	0.026
Neoabietic Acid	diss.	<0.001	<0.001	<0.001	<0.001	<0.001
	total	<0.001	<0.001	<0.001	<0.001	0.001

TABLE D8 CONCENTRATIONS OF POTENTIALLY TOXIC CONSTITUENTS IN INFLUENTS AND EFFLUENTS, October 15-19, 1979 AND INFLUENT SAMPLE COLLECTED DECEMBER 11, 1979, MILL 8

Compound	Date	Influent (mg/litre)					
		15.10.79	16.10.79	17.10.79	18.10.79	19.10.79	10.12.79
Juvabione/ Todomatuic Acid	diss.	16.8	13.0	9.4	11.0	6.8	6.0
	total	6.9	8.0	7.8	6.4	4.7	12.0
Linoleic Acid	diss.	0.76	1.3	1.1	0.96	1.1	1.6
	total	1.9	2.1	2.6	2.7	2.3	1.8
Oleic Acid	diss.	0.37	0.51	0.46	0.38	0.50	0.87
	total	0.97	1.1	1.3	1.3	1.2	0.96
Pimaric Acid/ Sandaracopimaric Acid	diss.	0.44	0.53	0.50	0.39	0.53	0.95
	total	0.33	0.86	0.85	0.73	0.60	1.3
Isopimaric Acid	diss.	2.3	3.5	2.7	2.4	2.9	4.4
	total	5.1	5.3	4.9	4.5	3.8	7.9
Levopimaric Acid	diss.	1.7	2.9	2.2	2.0	2.4	3.7
	total	4.2	4.4	4.0	3.9	3.0	6.7
Dehydroabietic Acid	diss.	1.7	2.5	1.9	1.6	2.1	4.1
	total	3.5	4.2	3.6	3.2	2.9	5.8
Abietic Acid	diss.	2.0	3.3	2.6	2.3	2.8	3.8
	total	4.7	4.9	4.5	4.1	3.5	6.8
Neoabietic Acid	diss.	0.49	1.0	0.77	0.64	0.73	1.2
	total	1.2	1.2	1.1	1.0	0.81	2.0

continued

TABLE D8(continued)

Compound	Date	Effluent (mg/litre)				
		15.10.79	16.10.79	17.10.79	18.10.79	19.10.79
Juvabione/ Todomatuic Acid	diss.	<0.001	<0.001	0.019	<0.001	<0.001
	total	0.030	0.018	0.086	0.12	0.13
Linoleic Acid	diss.	0.008	0.013	0.017	0.007	0.010
	total	0.039	0.16	0.13	0.22	0.27
Oleic Acid	diss.	0.015	0.022	0.054	0.017	0.022
	total	0.053	0.17	0.062	0.17	0.19
Pimaric Acid/ Sandaracopimaric Acid	diss.	<0.001	<0.001	<0.001	<0.001	<0.001
	total	<0.001	<0.001	0.003	<0.001	<0.001
Isopimaric Acid	diss.	<0.001	0.009	0.005	0.005	0.011
	total	0.026	0.026	0.065	0.034	0.049
Levopimaric Acid	diss.	<0.001	<0.001	<0.001	<0.001	<0.001
	total	<0.001	<0.001	<0.001	<0.001	<0.001
Dehydroabietic Acid	diss.	0.020	0.013	0.005	0.001	0.004
	total	0.037	0.041	0.018	0.017	0.022
Abietic Acid	diss.	<0.001	<0.001	<0.001	<0.001	<0.001
	total	0.011	0.030	0.012	<0.001	<0.001
Neoabietic Acid	diss.	<0.001	<0.001	<0.001	<0.001	<0.001
	total	<0.001	<0.001	<0.001	<0.001	<0.001

TABLE D9 RECOVERIES(%) OF COMPOUNDS ADDED TO INFLUENT AND EFFLUENT SAMPLES, WILLAMETTE AND ANDROSCOGGIN RIVER STUDIES

Compound	Sample [Mill, influent (Inf.), effluent (Eff.), date]									
	1 Inf. 19/10/79	3 Inf. 11/12/79	4 Inf. 17/10/79	4 Inf. 18/10/79	4 Eff. 18/10/79	4 Inf. 11/12/79	5 Inf. 18/10/79	5 Inf. 10/12/79	6 Inf. 11/12/79	7 Inf. 16/10/79
Trichloroguaiacol	100	90	103	80	190	80	100	90	85	120
Tetrachloroguaiacol	95	85	80	105	180	120	145	95	90	70
Juvabione/Todomatuic Acid	45	90	100	92	100	115	90	110	120	10
Linoleic Acid	95	95	85	76	100	75	95	85	95	95
Oleic Acid	85	80	56	50	70	50	75	85	95	70
Pimaric Acid/ Sandaracopimaric Acid	89	97	86	85	92	97	96	103	92	97
Isopimaric Acid	100	90	110	103	90	105	105	95	115	145
Levopimaric Acid	95	105	13	35	50	85	100	100	95	90
Dehydroabietic Acid	90	125	163	143	100	110	95	125	95	130
Abietic Acid	100	98	107	98	80	100	103	100	98	122
Neoabietic Acid	96	98	86	92	70	105	105	105	100	103
Monochlorodehydroabietic Acid	95	100	93	88	100	90	100	103	98	95
Dichlorodehydroabietic Acid	83	90	70	70	140	75	93	100	100	88

NA - not added

continued

TABLE D9(continued)

Compound	Sample [Mill, influent (Inf.), effluent (Eff.), date]							
	7 Eff. 17/10/79	7 Inf. 10/12/79	8 Inf. 11/12/79	I Inf. 7/2/80	I Eff. 8/2/80	II Eff. 6/2/80	IV Inf. 6/2/80	IV Eff. 7/2/80
Trichloroguaiacol	80	115	80	NA	83	80	150	90
Tetrachloroguaiacol	100	75	120	NA	90	120	140	90
Juvabione/Todomatuic Acid	170	125	115	100	100	80	90	90
Linoleic Acid	72	100	75	90	NA	90	110	80
Oleic Acid	70	90	50	60	NA	70	60	60
Pimaric Acid/ Sandaracopimaric Acid	91	125	97	90	82	90	82	94
Isopimaric Acid	90	105	105	80	80	100	90	90
Levopimaric Acid	20	110	85	110	30	70	30	100
Dehydroabietic Acid	100	75	110	120	100	100	100	110
Abietic Acid	80	125	100	90	80	90	110	90
Neoabietic Acid	15	137	105	105	85	85	100	95
Monochlorodehydroabietic Acid	90	130	90	85	95	95	90	95
Dichlorodehydroabietic Acid	80	132	75	55	55	70	60	60

NA - not added

TABLE D10 96-h LC50's OF PRIMARY-TREATED INFLUENTS DECEMBER 10,11, 1980 MEASURED BY BIOASSAY AND CALCULATED FROM ANALYSIS

Mill	96-h LC50* (% by vol.)			
	Total Toxicants		Dissolved Toxicants	
	Bioassay	Analysis	Bioassay	Analysis
1	15.8 (13.5 - 17.7)	10.8	19.2	17.5
2	94.4 (82.8 - 124)	NT†	NT	NT
3	9.5	19.6	23.3 (21.2 - 25.3)	43
4	12	11	19	19.6
5	45	100	45	~100
6	19.6 (17.3 - 21.8)	NT	27.2 (22.9 - 31.3)	NT
7	5.8 (5.3 - 6.3)	4.6	9.6 (8.8 - 11.3)	7.8
8	1.7 (1.60 - 1.86)	1.7	2.1	2.9

* 95% confidence limits in brackets
† NT - not toxic

TABLE D11 LOADINGS (Kg/day), WILLAMETTE RIVER MILLS

Compound		Mill 1		2		3		4	
		Influent	Effluent	Influent	Effluent	Influent	Effluent	Influent	Effluent
Trichloroguaiacol	Diss.	ND	ND	ND	ND	ND	ND	ND	ND
	Total								
Tetrachloroguaiacol	Diss.	ND	ND	ND	ND	ND	ND	ND	ND
	Total								
Juvabione/ Todomatuic Acid	Diss.		0.04		0.16		<0.04		0.23
	Total	21.7	0.08	0.41	0.58	4.87	<0.04	7.93	0.30
Linoleic Acid	Diss.		0.47		0.41		0.49		0.60
	Total	31.2	3.47	4.92	3.33	15.0	3.48	18.5	8.69
Oleic Acid	Diss.		0.83		0.90		0.80		0.76
	Total	16.2	1.50	3.28	<0.04	9.05	2.26	12.5	1.51
Pimaric/ Sandaracopimaric Acid	Diss.		<0.04		<0.04		<0.04		<0.04
	Total	10.6	0.04	<0.04	<0.04	9.75	1.05	10.2	0.98
Isopimaric Acid	Diss.		0.08		0.04		0.04		0.15
	Total	42.2	1.62	1.23	0.41	32.0	2.72	71.8	15.9
Levopimaric Acid	Diss.		<0.04		<0.04		<0.04		0.04
	Total	43.4	0.63	1.64	<0.04	28.9	0.70	40.4	3.89
Dehydroabietic Acid	Diss.		<0.04		0.04		<0.04		0.08
	Total	44.6	1.46	5.33	0.99	38.2	2.19	68.0	10.9
Abietic Acid	Diss.		0.04		<0.04		<0.04		0.04
	Total	46.9	1.58	2.87	0.33	28.2	1.53	71.8	15.1
Neoabietic Acid	Diss.		<0.04		<0.04		<0.04		<0.04
	Total	7.90	0.08	<0.04	<0.04	5.57	0.04	11.3	0.98

ND - not detected

continued

TABLE D11(continued)

Compound		Mill 5		Mill 6		Mill 7		Mill 8	
		Influent	Effluent	Influent	Effluent	Influent	Effluent	Influent	Effluent
Trichloroguaiacol	Diss.				1.83				
	Total	ND	ND	3.96	1.29	ND	ND	ND	ND
Tetrachloroguaiacol	Diss.				2.33				
	Total	ND	ND	7.43	0.64	ND	ND	ND	ND
Juvabione/	Diss.		0.23		0.40		<0.04		0.01
Todomatuic Acid	Total	3.46	<0.07	2.48	0.05	12.0	<0.04	8.08	0.10
Linoleic Acid	Diss.		0.90		0.45		0.33		0.01
	Total	30.8	5.95	4.95	1.09	56.2	14.1	2.73	0.19
Oleic Acid	Diss.		1.88		0.94		0.74		0.03
	Total	11.3	2.94	1.83	0.55	21.5	3.72	1.38	0.15
Pimaric/	Diss.		<0.07		<0.05		<0.04		<0.01
Sandaracopimaric Acid	Total	<0.07	<0.07	<0.05	<0.05	5.40	<0.04	0.80	<0.01
Isopimaric Acid	Diss.		0.07		0.05		0.08		0.01
	Total	2.85	0.38	4.95	0.05	57.0	1.53	5.58	0.05
Levopimaric Acid	Diss.		<0.07		<0.05		<0.04		<0.01
	Total	<0.07	<0.07	3.96	<0.05	50.8	0.33	4.63	<0.01
Dehydroabietic Acid	Diss.		0.07		<0.05		0.29		0.01
	Total	24.8	1.51	3.96	<0.05	53.5	1.03	4.16	0.03
Abietic Acid	Diss.		<0.07		<0.05		0.21		<0.01
	Total	3.61	0.45	1.00	<0.05	70.2	0.91	5.11	<0.01
Neoabietic Acid	Diss.		<0.07		<0.05		<0.04		<0.01
	Total	<0.07	<0.07	<0.05	<0.05	12.4	<0.04	1.26	<0.01

ND - not detected

TABLE D12 LOADINGS(g/tonne pulp), WILLAMETTE RIVER MILLS

Compound		Mill 1		2		3		4	
		Influent	Effluent	Influent	Effluent	Influent	Effluent	Influent	Effluent
Trichloroguaiacol	Diss.	ND	ND	ND	ND	ND	ND	ND	ND
	Total								
Tetrachloroguaiacol	Diss.	ND	ND	ND	ND	ND	ND	ND	ND
	Total								
Juvabione/ Todomatuic Acid	Diss.		0.08		1.14		<0.04		0.30
	Total	41.2	0.15	2.93	4.14	4.14	<0.04	10.4	0.39
Linoleic Acid	Diss.		0.89		2.93		0.49		0.79
	Total	59.2	6.58	35.1	23.8	12.7	3.46	24.3	11.4
Oleic Acid	Diss.		1.57		6.43		0.80		1.0
	Total	30.7	2.85	23.4	<0.29	7.69	2.25	16.4	1.98
Pimaric/ Sandaracopimaric Acid	Diss.		<0.07		<0.29		<0.04		<0.05
	Total	20.1	0.08	<0.29	<0.29	8.28	1.05	13.4	1.29
Isopimaric Acid	Diss.		0.15		0.29		0.04		0.20
	Total	80.1	3.07	8.78	2.93	27.2	2.71	94.2	20.9
Levopimaric Acid	Diss.		<0.07		<0.29		<0.04		0.05
	Total	82.3	1.19	11.7	<0.29	24.6	0.70	53.0	5.10
Dehydroabietic Acid	Diss.		<0.07		0.29		<0.04		0.10
	Total	84.6	2.77	38.1	7.07	32.5	2.18	89.2	14.3
Abietic Acid	Diss.		0.08		<0.29		<0.04		0.05
	Total	89.0	3.00	20.5	2.36	23.9	1.52	94.2	14.3
Neoabietic Acid	Diss.		<0.07		<0.29		<0.04		<0.05
	Total	15.0	0.15	<0.29	<0.29	4.73	0.04	14.8	1.29

ND - not detected

continued

TABLE D12(continued)

Compound		Mill 5 Influent	Mill 5 Effluent	Mill 6 Influent	Mill 6 Effluent	Mill 7 Influent	Mill 7 Effluent	Mill 8 Influent	Mill 8 Effluent
Trichloroguaiacol	Diss.				4.47				
	Total	ND	ND	9.25	3.15	ND	ND	ND	ND
Tetrachloroguaiacol	Diss.				5.69				
	Total	ND	ND	17.4	1.56	ND	ND	ND	ND
Juvabione/	Diss.		0.79		0.98		<0.06		0.04
Todomatuic Acid	Total	11.9	<0.24	5.79	0.12	19.5	<0.06	66.8	0.79
Linoleic Acid	Diss.		3.10		1.10		0.54		0.11
	Total	106	20.5	11.6	2.66	91.4	22.9	22.6	1.57
Oleic Acid	Diss.		6.48		2.30		1.20		0.26
	Total	38.8	10.1	4.27	1.35	34.9	6.05	11.4	1.27
Pimaric/	Diss.		<0.24		<0.12		<0.06		<0.01
Sandaracopimaric Acid	Total	<0.24	<0.24	<0.12	<0.12	8.8	<0.06	6.61	0.01
Isopimaric Acid	Diss.		0.24		0.12		0.13		0.06
	Total	9.79	1.31	11.6	0.12	92.7	2.49	46.1	0.40
Levopimaric Acid	Diss.		<0.24		<0.12		<0.06		<0.01
	Total	<0.24	<0.24	9.25	<0.12	82.6	0.54	38.3	<0.01
Dehydroabietic Acid	Diss.		0.24		<0.12		0.47		0.09
	Total	85.2	5.21	9.25	<0.12	86.7	1.67	34.4	0.26
Abietic Acid	Diss.		<0.24		<0.12		0.34		<0.01
	Total	12.4	1.55	2.34	0.12	114	1.48	42.2	0.01
Neoabietic Acid	Diss.		<0.24		<0.12		<0.06		<0.01
	Total	<0.24	<0.24	<0.12	<0.12	20.2	<0.06	10.4	<0.01

ND - not detected

TABLE E1 CONCENTRATIONS OF POTENTIALLY TOXIC COMPOUNDS IN INFLUENTS AND EFFLUENTS, MILL I
FEBRUARY 4-8, 1980

Compound		Influent (mg/l)						Effluent (mg/l)					
	Date	4/2	5/2	6/2	7/2	8/2	Mean	4/2	5/2	6/2	7/2	8/2	Mean
Trichloroguaiacol	Diss.	0.14	0.15	<0.01	0.28	<0.01	0.12	<0.001	0.026	0.098	0.089	0.011	0.045
	Total	0.22	0.34	0.18	0.19	<0.01	0.18	0.028	0.013	<0.001	0.008	0.007	0.011
Tetrachloroguaiacol	Diss.	<0.01	0.37	<0.01	0.41	0.26	0.26	[0.22]	<0.001	[0.17]	<0.001	<0.001	<0.001
	Total	<0.01	<0.01	0.43	<0.01	0.23	0.13	<0.001	<0.001	<0.001	0.033	0.023	0.011
Juvabione/ Todomatuic Acid	Diss.	0.05	0.06	0.05	0.08	0.04	0.06	0.007	0.026	0.005	0.009	<0.001	0.009
	Total	0.06	0.11	0.04	0.05	0.03	0.06	0.013	<0.001	0.006	0.015	0.011	0.009
Linoleic Acid	Diss.	2.0	3.5	2.5	2.3	1.9	2.4	0.034	0.040	0.021	0.026	0.023	0.029
	Total	3.0	4.5	2.9	3.3	2.2	3.2	0.11	0.18	0.15	0.20	0.23	0.17
Oleic Acid	Diss.	0.21	0.28	0.43	0.27	0.30	0.30	0.027	0.049	0.038	0.036	0.043	0.039
	Total	0.31	0.36	0.54	0.52	0.37	0.42	0.036	0.079	0.058	0.072	0.096	0.068
Pimaric/ Sandaracopimaric Acid	Diss.	0.09	0.14	0.17	0.16	0.09	0.13	0.021	0.018	<0.001	0.022	0.022	0.017
	Total	0.08	0.30	0.24	0.13	0.08	0.17	0.027	0.018	0.006	0.043	0.037	0.026
Isopimaric Acid	Diss.	0.32	0.51	0.82	0.75	0.78	0.64	0.048	0.038	0.021	0.11	0.066	0.056
	Total	0.37	0.69	1.0	0.87	0.74	0.73	0.042	0.052	0.030	0.12	0.069	0.063
Levopimaric Acid	Diss.	0.27	0.39	0.65	0.74	0.55	0.52	0.034	0.038	0.022	0.13	0.065	0.057
	Total	0.39	0.50	0.82	0.47	0.56	0.54	0.040	0.052	0.018	0.17	0.090	0.074
Dehydroabietic Acid	Diss.	0.24	0.35	0.47	0.48	0.49	0.40	0.015	0.038	0.018	0.048	0.032	0.030
	Total	0.41	0.46	0.44	0.45	0.38	0.43	0.014	0.021	0.029	0.067	0.044	0.035
Abietic Acid	Diss.	0.48	0.70	1.3	1.3	1.0	0.94	0.030	0.028	0.028	0.20	0.094	0.076
	Total	0.74	0.93	1.6	1.4	0.96	1.1	0.030	0.035	0.044	0.23	0.11	0.089
Neoabietic Acid	Diss.	0.40	0.65	0.68	1.2	0.61	0.70	0.005	0.026	0.024	0.087	0.063	0.041
	Total	0.46	0.91	0.90	0.63	0.49	0.68	0.007	0.019	<0.001	0.14	0.068	0.046
Monochlorodehydro-abietic Acid	Diss.	<0.01	0.11	0.10	0.16	<0.01	0.07	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
	Total	<0.01	0.14	0.12	0.15	<0.01	0.08	0.037	<0.001	0.025	0.060	0.044	0.035

[ ] - anomalous results omitted from calculation of mean value

TABLE E2 CONCENTRATIONS OF POTENTIALLY TOXIC COMPOUNDS IN INFLUENTS AND EFFLUENTS, MILL II

FEBRUARY 4-8, 1980

Compound		Influent (mg/l)						Effluent (mg/l)					
	Date	4/2	5/2	6/2	7/2	8/2	Mean	4/2	5/2	6/2	7/2	8/2	Mean
Trichloroguaiacol	Diss.	<0.01	ND	0.03	0.07	0.11	0.05	0.021	0.040	0.064	0.022	0.020	0.035
	Total	0.03	0.04	0.04	0.02	0.04	0.03	0.022	0.065	0.020	0.014	0.020	0.028
Tetrachloroguaiacol	Diss.	0.14	ND	0.01	<0.01	0.75	0.22	0.015	<0.001	0.11	0.043	<0.001	0.035
	Total	<0.01	<0.01	<0.01	0.16	0.32	0.10	0.019	0.046	0.022	0.010	0.096	0.039
Juvabione/	Diss.	0.44	ND	0.29	0.23	0.36	0.33	0.005	0.010	<0.001	0.005	0.006	0.005
Todomatuic Acid	Total	0.47	0.36	0.47	0.25	0.32	0.37	0.007	0.011	0.015	<0.001	0.012	0.011
Linoleic Acid	Diss.	8.4	ND	0.30	0.04	0.06	2.2	0.069	0.022	0.016	0.012	0.011	0.026
	Total	9.3	3.3	0.99	0.16	0.10	2.8	0.060	0.12	0.10	0.041	0.041	0.072
Oleic Acid	Diss.	0.54	ND	0.17	0.09	0.12	0.23	0.027	0.032	0.038	0.021	0.023	0.028
	Total	0.90	0.66	0.32	0.12	0.11	0.42	0.029	0.063	0.075	0.055	0.040	0.052
Pimaric/	Diss.	0.32	ND	0.04	0.02	0.08	0.12	0.004	<0.001	<0.001	0.002	0.003	0.002
Sandaracopimaric Acid	Total	0.47	0.14	0.07	0.05	0.06	0.16	0.002	0.012	0.006	<0.001	<0.001	0.004
Isopimaric Acid	Diss.	1.6	ND	0.16	0.16	0.23	0.54	0.020	0.011	0.008	0.009	0.004	0.010
	Total	1.9	0.65	0.36	0.20	0.27	0.68	0.019	0.069	0.040	0.017	0.009	0.031
Levopimaric Acid	Diss.	1.4	ND	0.15	0.19	0.17	0.48	0.019	0.002	0.004	0.009	<0.001	0.007
	Total	1.1	0.54	0.28	0.27	0.19	0.48	0.010	0.063	0.020	0.004	<0.001	0.019
Dehydroabietic Acid	Diss.	1.2	ND	0.26	0.53	0.50	0.62	0.014	0.014	0.006	0.007	0.004	0.009
	Total	1.7	0.59	0.58	0.55	0.48	0.77	0.016	0.035	0.031	<0.001	0.011	0.019
Abietic Acid	Diss.	3.0	ND	0.24	0.34	0.40	1.0	0.028	0.011	0.011	0.015	0.006	0.014
	Total	3.4	1.0	0.48	0.41	0.44	1.2	0.019	0.077	0.053	0.026	0.013	0.038
Neoabietic Acid	Diss.	0.99	ND	0.13	0.11	0.11	0.33	0.007	<0.001	<0.001	<0.001	<0.001	0.001
	Total	0.88	0.32	0.22	0.15	0.14	0.34	0.004	0.019	<0.001	<0.001	0.023	0.009
Monochlorodehydro-	Diss.	<0.01	<0.01	0.08	0.04	0.14	0.05	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
abietic Acid	Total	0.07	<0.01	0.12	0.04	0.05	0.06	0.005	0.019	0.012	<0.001	<0.001	0.007

ND - not determined

TABLE E3 CONCENTRATIONS OF POTENTIALLY TOXIC COMPOUNDS IN INFLUENTS AND EFFLUENTS, MILL III

FEBRUARY 4-8, 1980

Compound	Date	Influent (mg/l)						Effluent (mg/l)					
		4/2	5/2	6/2	7/2	8/2	Mean	4/2	5/2	6/2	7/2	8/2	Mean
Juvabione/ Todomatuic Acid	Diss.	0.61	0.57	0.56	0.78	0.71	0.65	0.008	0.003	<0.001	0.029	<0.001	0.008
	Total	0.75	0.52	0.58	0.83	0.88	0.71	0.005	0.010	0.008	0.025	0.033	0.016
Linoleic Acid	Diss.	<0.01	0.04	0.02	0.04	0.05	0.03	0.015	0.029	0.019	0.020	0.012	0.019
	Total	2.3	1.1	1.4	1.9	2.2	1.8	0.026	0.099	0.023	0.043	0.088	0.056
Oleic Acid	Diss.	0.03	0.06	0.05	0.05	0.05	0.05	0.028	0.033	0.049	0.024	0.024	0.032
	Total	0.65	0.31	0.47	0.46	0.53	0.48	0.024	0.044	0.038	0.035	0.048	0.038
Pimaric/ Sandaracopimaric Acid	Diss.	<0.01	<0.01	<0.01	0.01	<0.01	<0.01	<0.001	<0.001	<0.001	0.002	<0.001	<0.001
	Total	0.22	0.17	0.29	0.30	0.27	0.25	0.012	0.003	<0.001	0.005	0.014	0.007
Isopimaric Acid	Diss.	<0.01	<0.01	0.02	0.01	0.01	0.01	0.010	0.005	0.003	0.008	0.008	0.007
	Total	1.1	0.66	0.74	0.96	1.2	0.93	0.037	0.027	0.012	0.036	0.11	0.045
Levopimaric Acid	Diss.	<0.01	<0.01	0.04	0.41	0.01	0.09	0.008	<0.001	<0.001	0.006	0.005	0.004
	Total	1.4	0.74	0.93	1.9	2.8	1.5	0.045	0.037	0.011	0.039	0.11	0.048
Dehydroabietic Acid	Diss.	0.06	0.12	0.20	0.07	<0.01	0.09	0.009	0.006	0.004	<0.001	0.005	0.005
	Total	0.85	0.78	0.74	0.77	0.64	0.75	0.063	0.055	0.008	0.020	0.066	0.042
Abietic Acid	Diss.	0.02	<0.01	0.07	0.04	0.01	0.03	0.009	<0.001	0.004	0.011	0.010	0.007
	Total	2.1	1.2	1.6	1.8	2.1	1.8	0.088	0.051	0.014	0.052	0.18	0.076
Neoabietic Acid	Diss.	<0.01	<0.01	0.02	<0.02	0.01	<0.01	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
	Total	0.41	0.12	0.35	0.45	0.55	0.38	0.019	<0.001	<0.001	0.015	0.039	0.015

TABLE E4 CONCENTRATIONS OF POTENTIALLY TOXIC COMPOUNDS IN INFLUENTS AND EFFLUENTS, MILL IV

FEBRUARY 4-8, 1980

Compound		Influent (mg/l)						Effluent (mg/l)					
	Date	4/2	5/2	6/2	7/2	8/2	Mean	4/2	5/2	6/2	7/2	8/2	Mean
Trichloroguaiacol	Diss.	0.04	0.17	0.03	0.22	<0.01	0.10	0.032	0.056	0.131	0.013	0.005	0.047
	Total	0.04	0.23	0.12	<0.01	0.13	0.11	0.024	0.027	0.061	0.060	0.004	0.042
Tetrachloroguaiacol	Diss.	<0.01	<0.01	<0.01	0.23	0.21	0.09	0.053	<0.001	[0.336]	0.014	<0.001	0.013
	Total	0.03	<0.01	<0.01	0.02	0.37	0.08	0.012	0.039	<0.001	0.053	<0.001	0.026
Juvabione/ Todomatuic Acid	Diss.	0.39	1.1	0.23	0.15	0.08	0.39	0.20	0.27	0.077	0.028	0.006	0.12
	Total	0.31	0.56	0.25	0.16	0.07	0.27	0.23	0.17	0.083	0.033	0.006	0.10
Linoleic Acid	Diss.	1.8	0.14	0.97	0.10	0.11	0.63	0.034	0.025	0.031	0.037	0.044	0.034
	Total	2.4	0.29	1.5	0.33	0.22	0.96	0.074	0.059	0.059	0.078	0.044	0.063
Oleic Acid	Diss.	0.34	0.09	0.29	0.10	0.04	0.17	0.042	0.042	0.043	0.049	0.051	0.045
	Total	0.54	0.18	0.43	0.18	0.14	0.29	0.063	0.046	0.052	0.062	0.048	0.054
Pimaric/ Sandaracopimaric Acid	Diss.	0.18	0.10	0.12	0.06	0.10	0.11	0.023	0.020	0.018	0.019	0.010	0.018
	Total	0.20	0.14	0.14	0.10	0.13	0.14	0.036	0.026	0.023	0.024	0.012	0.024
Isopimaric Acid	Diss.	1.0	0.26	0.51	0.22	0.39	0.48	0.088	0.056	0.071	0.075	0.031	0.064
	Total	1.1	0.41	0.63	0.31	0.50	0.59	0.15	0.10	0.10	0.089	0.025	0.094
Levopimaric Acid	Diss.	0.73	0.32	0.37	0.22	0.26	0.38	0.15	0.079	0.11	0.11	0.037	0.097
	Total	0.73	0.41	0.39	0.19	0.30	0.41	0.20	0.14	0.14	0.083	0.033	0.12
Dehydroabietic Acid	Diss.	0.66	0.43	0.46	0.28	0.40	0.45	0.15	0.13	0.11	0.072	0.026	0.098
	Total	0.68	0.50	0.51	0.32	0.47	0.50	0.21	0.13	0.13	0.087	0.023	0.12
Abietic Acid	Diss.	1.4	0.49	0.72	0.41	0.75	0.76	0.23	0.16	0.16	0.16	0.047	0.15
	Total	1.4	0.68	0.82	0.49	0.87	0.86	0.35	0.22	0.20	0.16	0.040	0.20
Neoabietic Acid	Diss.	0.57	0.51	0.34	0.20	0.17	0.36	0.080	0.026	0.053	0.044	0.014	0.043
	Total	0.47	0.38	0.31	0.15	0.16	0.29	0.12	0.072	0.038	0.022	0.013	0.054
Monochlorodehydro-abietic Acid	Diss.	0.10	<0.01	0.07	<0.01	<0.01	0.03	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
	Total	0.11	0.13	0.08	0.09	<0.01	0.08	<0.001	<0.001	<0.001	0.029	0.006	0.007

[ ] - anomalous results omitted from calculation of mean value

TABLE E5 CONCENTRATIONS OF TOXIC COMPOUNDS
IN GRAB SAMPLES OF INFLUENT COLLECTED FOR BIOASSAY FEBRUARY 7,8, 1980

Compounds	Total Toxicants (mg/1)							
	Mill I		Mill II		Mill III		Mill IV	
	7/2	8/2	7/2	8/2	7/2	8/2	7/2	8/2
Trichloroguaiacol	0.18	0.23	0.04	0.04	<0.01	<0.01	<0.01	0.05
Tetrachloroguaiacol	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.21	0.12
Juvabione/Todomatuic Acid	0.04	0.05	0.18	0.21	0.23	0.49	0.03	0.19
Linoleic Acid	3.2	1.9	0.93	0.22	9.0	2.5	1.2	4.3
Oleic Acid	0.83	0.35	0.41	0.18	3.0	0.76	0.29	1.9
Pimaric/Sandaracopimaric Acid	0.30	0.09	0.08	0.05	1.2	0.23	0.10	0.52
Isopimaric Acid	1.3	0.87	0.35	0.30	5.8	1.8	0.52	3.1
Levopimaric Acid	1.7	0.64	0.03	0.27	6.5	1.8	0.38	2.4
Dehydroabietic Acid	0.82	0.57	1.0	0.87	2.7	1.1	0.51	1.1
Abietic Acid	2.6	1.1	0.48	0.42	10.0	3.3	0.78	4.8
Neoabietic Acid	1.0	0.59	0.09	0.10	2.7	0.68	0.12	0.78
Monochlorodehydroabietic Acid	0.10	0.13	0.11	0.14	<0.01	<0.01	<0.01	0.10
Total Toxic Units	13.3	7.3	2.6	2.5	49.9	15.1	4.5	22.5

continued

TABLE E5(continued)

Compounds	Dissolved Toxicants (mg/l)							
	Mill I		Mill II		Mill III		Mill IV	
	7/2	8/2	7/2	8/2	7/2	8/2	7/2	8/2
Trichloroguaiacol	<0.01	0.16	0.02	0.04	<0.01	<0.01	<0.01	0.04
Tetrachloroguaiacol	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.11
Juvabione/Todomatuic Acid	0.02	0.02	0.18	0.17	0.69	0.52	0.04	0.09
Linoleic Acid	2.2	0.99	0.37	0.10	0.21	0.09	0.21	2.0
Oleic Acid	0.50	0.24	0.21	0.12	0.08	0.05	0.12	0.83
Pimaric/Sandaracopimaric Acid	0.16	0.06	<0.01	<0.01	<0.01	<0.01	0.09	0.40
Isopimaric Acid	0.82	0.50	0.13	0.17	0.09	0.02	0.43	2.2
Levopimaric Acid	1.0	0.38	<0.01	0.15	0.09	<0.01	0.29	1.6
Dehydroabietic Acid	0.62	0.39	0.38	0.65	0.10	0.09	0.57	1.0
Abietic Acid	1.6	0.63	0.18	0.23	0.11	0.03	0.59	3.3
Neoabietic Acid	0.66	0.38	0.05	0.08	0.01	<0.01	0.12	0.54
Monochlorodehydroabietic Acid	0.09	0.07	0.06	0.08	<0.01	<0.01	<0.01	0.15
Total Toxic Units	8.0	3.9	0.8	1.3	0.8	0.4	2.6	16.3

TABLE E6 COMPARISON OF 96-h LC50's MEASURED BY BIOASSAY AND CALCULATED FROM ANALYSIS

Mill	Date	96-h LC50* (% by vol.)					
		Total Toxicants				Dissolved Toxicants	
		Influent		Effluent		Influent	
		Measured	Analysis	Measured	Analysis	Measured	Analysis
I	7/2/80	12.2	7.5	NT	NT	15.1 (13.9-17.1)	12.5
	8/2/80	17.3	14.7	NT	NT	27	26
II	7/2/80	37.6 (30.3-70.9)	38.5	NT	NT	77 (48.6-100)	NT
	8/2/80	35.6 (28.9-58)	40	NT	NT	41	77
III	7/2/80	5.6 (4.79-6.18)	2.0	NT	NT	15.3 (13.7-17.6)	NT
	8/2/80	7.0 (5.91-7.99)	6.6	NT	NT	8.4	NT
IV	7/2/80	37.5 (29.7-94.3)	25	NT	NT	NT	38.5
	8/2/80	5.4 (4.8-6.0)	4.4	NT	NT	4.9 (4.22-5.72)	6.1

* 95% confidence limits in brackets
NT not toxic

TABLE E7 LOADINGS (Kg/day), ANDROSCOGGIN RIVER MILLS

Compound		Mill I Influent	Mill I Effluent	Mill II Influent	Mill II Effluent	Mill III Influent	Mill III Effluent	Mill IV Influent	Mill IV Effluent
Trichloroguaiacol	Diss.		3.60		6.22		ND		5.02
	Total	14.4	0.88	5.33	5.00	ND	ND	11.8	4.49
Tetrachloroguaiacol	Diss.		<0.08		6.22		ND		1.39
	Total	10.4	0.88	17.8	6.93	ND	ND	8.55	2.78
Juvabione/ Todomatuic Acid	Diss.		0.72		0.89		0.09		12.4
	Total	4.73	0.72	66.5	1.95	8.0	0.18	28.9	11.0
Linoleic Acid	Diss.		2.32		4.62		0.21		3.63
	Total	256	14.0	492	12.8	20.1	0.63	102	6.73
Oleic Acid	Diss.		3.12		4.98		0.36		4.81
	Total	33.6	5.45	74.8	9.24	5.44	0.43	31.0	5.77
Pimaric/ Sandaracopimaric Acid	Diss.		1.12		0.36		<0.01		1.92
	Total	13.4	2.08	28.1	0.89	2.78	0.08	15.0	2.57
Isopimaric Acid	Diss.		4.49		1.78		0.08		6.84
	Total	58.5	5.05	120	5.51	10.5	0.51	63.1	10.0
Levopimaric Acid	Diss.		4.56		1.24		0.05		10.4
	Total	43.5	5.93	85.6	3.38	17.4	0.54	43.8	12.8
Dehydroabietic Acid	Diss.		2.40		1.60		0.06		10.5
	Total	34.4	2.80	137	3.38	8.51	0.47	53.5	12.4
Abietic Acid	Diss.		6.09		2.49		0.08		15.8
	Total	90.2	7.13	205	6.75	19.9	0.86	91.9	20.7
Neoabietic Acid	Diss.		3.28		0.18		<0.01		4.60
	Total	54.4	3.68	60.4	1.60	4.26	0.17	31.0	5.77
Monochlorodehydro-abietic Acid	Diss.		<0.02		<0.18		ND		<0.11
	Total	1.69	0.74	10.7	1.24	ND	ND	8.55	0.75

TABLE E8 LOADINGS (g/tonne pulp), ANDROSCOGGIN RIVER MILLS

Compound		Mill I		Mill II		Mill III		Mill IV	
		Influent	Effluent	Influent	Effluent	Influent	Effluent	Influent	Effluent
Trichloroguaiacol	Diss.		3.72		5.27		ND		8.44
	Total	14.9	0.91	4.51	4.23	ND	ND	19.8	7.55
Tetrachloroguaiacol	Diss.		<0.08		5.27		ND		2.32
	Total	10.8	0.91	15.1	5.87	ND	ND	14.4	4.67
Juvabione/ Todomatuic Acid	Diss.		0.74		0.75		0.69		20.8
	Total	4.89	0.74	56.3	1.65	61.9	1.39	48.6	18.5
Linoleic Acid	Diss.		2.40		3.91		1.62		6.1
	Total	265	14.5	416	10.8	155	4.86	171	11.3
Oleic Acid	Diss.		3.23		4.22		2.78		8.08
	Total	34.6	5.64	63.3	7.82	41.9	3.32	52.1	9.70
Pimaric/ Sandaracopimaric Acid	Diss.		1.16		0.30		<0.08		3.23
	Total	13.9	2.15	23.8	0.75	21.4	0.62	25.2	4.32
Isopimaric Acid	Diss.		4.64		1.51		0.62		11.5
	Total	60.5	5.22	102	4.66	81.0	3.93	106	16.8
Levopimaric Acid	Diss.		4.72		1.05		0.39		17.5
	Total	45.0	6.13	72.5	2.86	134	4.16	73.6	21.5
Dehydroabietic Acid	Diss.		2.48		1.35		0.46		17.6
	Total	35.6	2.90	116	2.86	65.6	3.62	89.9	20.8
Abietic Acid	Diss.		6.30		2.11		0.62		26.6
	Total	93.3	7.37	174	5.71	153	6.63	154	34.8
Neoabietic Acid	Diss.		3.39		0.15		<0.08		7.73
	Total	56.3	3.81	51.1	1.35	32.8	1.31	52.1	9.70
Monochlorodehydro-abietic Acid	Diss.		<0.02		<0.15		ND		<0.18
	Total	1.75	0.76	9.06	1.05	ND	ND	14.4	1.26

APPENDIX F

WILLAMETTE RIVER

Total volume of effluent discharged by pulp mills to Willamette River was 3.82 m^3/sec.

Willamette River flow at USGS gauging station 14211720, Portland, OR., during the sampling period, October 15-19, 1979 averaged 356 m^3/sec. Thus, total dilution available was 93-fold.

Dilutions available at upstream USGS gauging stations 14191000 (Salem), 14174000 (Albany), and 14166000 (Harrisburg) were 126-fold, 137-fold and 180-fold, respectively, taking into account river flow and effluent volumes discharged from mills above each station.

Average flow of the Willamette River at Portland is 935 m^3/sec during the period of record (1972-1979). Total dilution available for pulp mill effluents would be 245-fold, compared with 277-fold at Salem, 264-fold at Albany and 360-fold at Harrisburg for the corresponding long-term means and appropriate effluent discharge volumes.

Minimum flow of the Willamette River at Portland during the period of record is 119 m^3/sec, in 1978. Total dilution available for pulp mill effluents would be 31-fold, compared with 30-fold at Salem (minimum in 1940), 34-fold at Albany (minimum in 1940), and 58-fold at Harrisburg (minimum in 1944).

ANDROSCOGGIN RIVER

Total volume of effluent discharged by pulp mills to Androscoggin River was 4.34 m^3/sec.

Androscoggin River flow at USGS gauging station 01059000, Auburn, ME., during sampling period, February 4-8, 1980, averaged 75 m^3/sec. Thus, total dilution available was 17-fold.

Dilutions available at upstream USGS gauging stations 01054500 (Rumford) and 01054000 (Gorham) were 25 times, and 59 times, respectively, taking into account river flow and effluent volume discharged from mills above each station.

Average flow of the Androscoggin River at Auburn during the period of record (1928-1979) is 175 m^3/sec. Total dilution available for pulp mill effluents would be 40-fold, compared with 49-fold at Rumford and 76-fold at

Gorham for the corresponding long-term means and appropriate discharge volumes.

Minimum flow of the Androscoggin River at Auburn during the period of record in 9.6 m^3/sec in 1941. Total dilution available for pulp mill effluents under this condition would be 2.2-fold, compared with 8-fold at Rumford (minimum in 1911) and 24-fold at Gorham (minimum in 1948).